ANALYTICAL ABSTRACTS

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Editor: NORMAN EVERS, B.Sc., Ph.D., F.R.I.C.
20, EASTCHEAP, LONDON, E.C.3
Telephone: MANsion House 6608

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

874. [**Review of progress in analytical chemistry.**] (*Anal. Chem.*, 1954, **26** [1], 2–181).—An annual review of progress in analytical chemistry under the following 25 headings is given by different authors: light absorption spectrometry, infra-red spectroscopy, ultra-violet absorption spectrophotometry, X-ray absorption and emission, X-ray diffraction, electron microscopy, chemical microscopy, Raman spectroscopy, emission spectroscopy, mass spectrometry, organic polarography, acid - base titrations in non-aqueous solvents, potentiometric titrations, chromatography, distillation analysis, ion exchange, extraction, organic microchemistry, inorganic microchemistry, fluorimetric analysis, electroanalysis, inorganic gravimetric analysis, volumetric analytical methods for organic compounds, biochemical analysis and nucleonics.
N. E.

875. **Standardisation of normal acid solutions.** A. Desjobert and F. Petek (*Anal. Chim. Acta*, 1954, **10** [1], 10–22).—The principal methods used for standardisation of acid soln., *viz.*, the weighing of a salt of the acid or titration with a standard alkali soln., are critically reviewed. The use of K_2CO_3, prepared by calcining pure commercial $KHCO_3$ is suggested for the preparation of a standard alkali soln. An Erlenmeyer flask fitted with a ground-in stopper is tared after heating on a sand-bath for 30 min. at 300° to 350° C, whilst the stopper is kept separately in an oven at 100° C. Both are cooled in a desiccator, which should contain conc. H_2SO_4. $KHCO_3$ (2·2 to 2·3 g) is weighed into the flask, which is heated as before for 2 hr. The wt. of K_2CO_3 is found on re-weighing. Ten ml of dist. H_2O are added, and when most of the solid has dissolved, 20 ml of the acid soln. are slowly introduced from a pipette and the soln. is shaken. After addition of 2 drops of an indicator containing 0·050 g of methyl orange and 0·250 g of bromocresol green in 50 ml of absolute alcohol, more acid is run in from a burette until the indicator turns from blue through a yellow-green to a champagne tint. If the mean of 2 results is taken, the precision of the standardisation is about ±0·2 per cent.
J. H. Waton

876. **Microchemistry applied to chemical analysis.** T. S. West (*Research*, 1954, **7** [2], 60–67).—The special methods and apparatus required in the technique of micro-analysis, in which sample weights are restricted to the order of 0·1 to 5 mg, are reviewed. The operations and determinations used in both inorganic and organic qual. and quant. analyses are dealt with. Reference is made to ultramicro-techniques, involving sample weights of 10^{-4} to 10^{-7} g.
G. C. Jones

877. **Potentiometric interpretation of some volumetric methods.** F. Sierra and O. Carpena (*An. Soc. Esp. Fis. Quim., B*, 1953, **49** [12], 769–772).—During titration of NaCl with $AgNO_3$, the pH rises steadily until the end-point, when it drops suddenly. In NaBr - $AgNO_3$ and NaI - $AgNO_3$ titrations, the pH remains approx. const. until a sudden fall at the end-point. During titration of NaBr with $AgClO_4$, the pH falls steadily, but more rapidly at the end-point. These findings are in accordance with the expected adsorption of ions by the ppt.
D. P. Young

878. **Differential method for precision colorimetric analysis.** A. Ringbom and K. Österholm (*Anal. Chem.*, 1953, **25** [12], 1798–1803).—A differential method for colorimetric analysis, which utilises the sensitivity of photo-electric instruments more effectively than is usual, is described. The method avoids errors caused by imperfect construction and positioning of the cuvettes, and standard solutions of high absorbance are not required. The precision and the most favourable experimental conditions are derived by theoretical considerations and these give satisfactory results, even if Beer's law is not valid, provided the standards are suitably chosen. The method when applied to the determination of $Cu(ClO_4)_2$ and Fe - *o*-phenanthroline solutions is accurate to within 1 or 2 parts per 1000.
G. P. Cook

879. **Paper chromatography: its principles and uses.** H. Weil (*Canad. Chem. Processing*, 1954, **38** [1], 68, 70 and 72).—The main events in the evolution of paper chromatography are reviewed.
G. C. Jones

880. **A simple method of making transfers in paper chromatography.** A. M. Moore and J. B. Boylen (*Science*, 1953, **118**, 19–20).—An arrangement is illustrated by which water-sol. compounds can be transferred from one paper strip to another for development with a different solvent, while keeping the spot on the second strip small enough for development. This is done by blowing a stream of air at room temp. on to the under side of the second strip so that the water evaporates as quickly as the eluate flows.
N. E.

881. **A new paper-chromatographic technique for the detection and identification of inorganic ions.** S. N. Tewari (*Kolloidzschr.*, 1953, **133** [2–3], 132).—The ascending technique of solvent flow on a paper strip is modified by fixing several horizontal strips at equidistant spaces from the bottom. Advantages are simplicity and compactness; bands are clear and reproducibility for metallic cations is good.
A. B. Densham

882. **Paper-chromatographic separation of hydrophobic substances with acetylated cellulose paper.** F. Micheel and H. Schweppe (*Mikrochim. Acta*, 1954, [1], 53–63).—Filter-paper that has been acetylated while retaining its fibre structure can be used for the paper chromatographic separation of strongly or partially hydrophobic materials. The method is effective for the separation and detection of sugar acetates, isomeric phenols and isomeric aromatic

amines or their derivatives, or hydrophobic dyestuffs, such as those used for the dyeing of acetate rayon. A. J. MEE

883. **Estimation of sample weight for absorption spectra determinations.** F. S. Boig (*Chemist Analyst*, 1953, **42** [4], 86–87).—A method is described for the estimation of sample weights in the u.v. absorption spectra study of a number of closely related compounds. By use of the equation applicable to u.v. absorption determinations $\left(\epsilon = \frac{MA}{bc}\right)$ and by assuming a cell light-path of 1 cm and an absorbance of 0·800, a table of sample weights can be constructed for a mol. wt. range of 50 to 400 and log ϵ of 1·00 to 5·00. Values for other absorbances and other lengths of cell path can be obtained by using appropriate factors. D. BAILEY

884. **Infra-red spectroscopy.** W. G. Wearmouth (*Lab. Practice*, 1953, **2** [6, 7 and 8], 297–300; 372–376 and 422–427).—Advances in techniques during the past 15 years are reviewed. R. B. CLARKE

885. **Recent advances in infra-red spectroscopy.** J. K. Brown (*B.C.U.R.A. Mon. Bull.*, 1953, **17**, 449–464).—A critical review (232 references.) R. B. CLARKE

886. **The vacuum fusion method for determining minute quantities of gases in metals.** W. J. McMahon and L. S. Foster (*J. Chem. Educ.*, 1953, **30** [12], 609–613).—Principles, scope and accuracy of the vacuum fusion method for determination of minute quantities of gases in metals are discussed, and design of apparatus and its manipulation are described. D. A. PANTONY

887. **Some applications of differential thermal analysis and findings using this technique.** T. L. Webb (*S. Afr. Ind. Chem.*, 1953, **7** [12], 224–232).—The general principles of differential thermal analysis are discussed, and its application in various branches of applied chemistry is briefly mentioned. Work carried out on hydrated dolomitic limes is discussed in some detail, with particular reference to the interpretation of thermograms. A bibliography is given. A. WEBSTER

888. **Magnetic and thermomagnetic analysis.** G. Guiot-Guillian (*Chim. Anal.*, 1954, **31** [6], 9–17).—A review with 29 references. E. HAYES

889. **Chemical analysis by means of high frequency oscillators.** P. W. West (*Selecta Chim., S. Paulo*, 1953, [12], 19–35).—The use of high-frequency oscillators for the chemical analysis of organic and inorganic compounds in aq. and non-aq. systems is reviewed. The theoretical concepts of high-frequency techniques are outlined and the developments in instrument design are discussed. Two groups of instruments are described, one includes instruments that measure the response derived from the chemical systems in terms of current or voltage effects and the other measures the frequency changes induced by diverse chemical systems. The application of the method to titrimetric analyses, direct concn. measurements (including binary systems), following of high speed reactions, complex formation and in locating chromatographic zones is described. Future developments are discussed. D. BAILEY

890. **A stable form of methyl orange - indigo carmine indicator.** H. J. Saling (*Chemist Analyst*, 1953, **42** [4], 87–88).—A stable and convenient form of methyl orange - indigo carmine mixed indicator is obtained by impregnating filter-paper with a freshly prepared soln. (1 g of methyl orange and 3 g of indigo carmine in 1 litre of soln.) and drying at 60° C. A strip of the indicator paper is immersed in the soln. to be titrated and the indicator is leached from the paper. The end-point obtained by the use of this indicator paper is of the same colour and of equal sharpness as that obtained by the use of freshly prepared indicator soln. The paper gives good results when stored for more than 6 months. D. BAILEY

891. **Dry reagents for analytical purposes. Mixtures.** E. Dannenberg (*Anal. Chim. Acta*, 1954, **10** [2], 101–107).—The principles governing the preparation dry mixtures for analytical work are discussed and a classification is made according to their stability and use. Examples given are Benedict's reagent and the Folin - Wu copper reagent; it is suggested that the more soluble $CuCl_2$ and more soluble complexing reagents might be substituted in these. Rothera's reagent does not keep well and it is suggested that a reagent A (10 per cent. nitroprusside - 90 per cent. NaCl) and a reagent B [50 g of $(NH_4)_2SO_4$ and 50 g of Na_2CO_3] be used (in the proportion 1 : 10) with the possible substitution of sodium phosphate for Na_2CO_3 so that reagent B does not smell of ammonia. A reagent containing *o*-tolidine (1 pt.), tartaric acid (3 pt.), BaO_2 (3 pt.) and Ca acetate (3 pt.) is described as a substitute for benzidine reagents for catalysts. A reagent containing 1-naphthylamine (0·1 g), sulphanilic acid (0·5 g) and tartaric aid (6 g) for nitrites is given and it is noted that naphthylamine acetate is not a suitable substitute. This reagent has been kept unchanged for 4 years. A dry form of the Folin - Wu molybdotungstophosphate reagent is described. The use of NaCl mixed with a little dyestuff for density measurements is suggested. E. J. H. BIRCH

See also Abstract 956.

2.—INORGANIC ANALYSIS

892. **Precision determinations of deuterium in aqueous solution by a pyknometer method.** L. Silverman and W. Bradshaw (*Anal. Chim. Acta*, 1954, **10** [1], 68–77).—The determinations of ^2H content are carried out with two calibrated 50-ml Boots pyknometers, which form a matched pair, by a double-weighing technique. Over the whole range 1 to 99 atom per cent. ^2H, the average deviation is $\pm$ 0·02 atom per cent. ^2H. Where possible, the sample should be compared with a standard of approx. the same ^2H content, made by mixing H_2O with an analysed deuterium oxide standard high ^2H content. When this is not convenient, the standard is used for the 75 to 100 atom per cent. samples, distilled water is used for those in the 0 to 25 atom per cent. range, and an intermediate standard is used for samples in the 25 to 75 atom per cent. range. J. H. WATON

893. **Separation and identification of alkali metals on paper chromatograms.** A. E. Steel (*Nature*, 1954, **173**, 315–316).—A method has been developed depending on the production of an insoluble coloured ppt. when K^+, Cs^+, Rb^+ and NH_4^+ are treated with sodium cobaltinitrite soln. The soln. of Kramer and Tisdall (*J. Biol. Chem.*, 1921, **46**, 339) or a freshly prepared 10 per cent. aq. soln. of sodium cobaltinitrite in 5 per cent. acetic acid can be used. N. E.

894. **Flame spectrophotometric determination of sodium and potassium in viscous solutions or plant extracts.** H. M. Bauserman and R. R. Cerney, jun. (*Anal. Chem.*, 1953, **25** [12], 1821–1824).—Na and K are determined by a flame-spectrophotometric method, LiCl being used as an internal standard to overcome errors due to the variable physical characteristics of the samples analysed. The correction factor for these characteristics is calculated from the ratio of the Li standard added to the Li found from the transmittance readings. The best results are obtained in the concentration range of 50 to 500 p.p.m. and the largest deviation is $\simeq$ 10 per cent., other results giving a deviation of 1·6 to 5·3 per cent. G. P. Cook

895. **Improvements in flame-photometric determination of sodium in Portland cement.** J. J. Diamond and L. Bean (*Anal. Chem.*, 1953, **25** [12], 1825–1830).—Improvements in the flame-photometric method for the determination of Na in Portland cement are recommended. Some flame photometers were found consistently to indicate a Na content of less than zero for certain cements, and, in addition, different photometers hardly ever gave identical results for any cement. Improvement is effected by removal of SiO_2 from the cement by usual means before the determination of Na at 589 mμ. Further improvement is achieved by narrowing the effective band width of the instrument by introducing to its optical system a multilayer inteference filter. On incorporating these modifications there is close agreement with the J. Lawrence Smith gravimetric method for Na. G. P. Cook

896. **Radiometric determination of small amounts of potassium.** T. Ishimori and Y. Takashima (*Bull. Chem. Soc. Japan*, 1953, **26** [9], 481–484).—Experiments to obtain a working relationship between concn. of small amounts of K and the relative intensity of radioactivity obtained with solutions of $Na_3Co(NO_2)_6$ labelled with ^{60}Co as the precipitant, having various specific activities, and standard KCl solutions are described. A near-linear relationship is found for K concentrations of 8 to 0·3 and 0·126 to 0·0016 mg, but there is deviation from linearity with 0·8 to 0·16 mg. A comparison of results is made with gravimetric analysis on samples of andesite. D. E. Blenford

897. **Analysis for industry. Determination of potassium by precipitation as potassium tetraphenylboron. I.** A. J. Nutten (*Ind. Chem.*, 1954, **30**, 25–28).—A review with 13 references of the use of Li and Na tetraphenylborons for the gravimetric determination of K. Soc. Chem. Ind. Abstr.

898. **Determination of copper in OFHC [oxygen-free high-conductivity] copper.** J. Kinnunen *Chemist Analyst*, 1953, **42** [4], 93–94).—A combined electrolytic - colorimetric method is described for the determination of Cu in oxygen-free high-conductivity Cu. The Cu borings (10 g) are dissolved in 90 ml of acid mixture consisting of 750 ml of H_2O, 250 ml of conc. HNO_3 and 1000 ml of dil. H_2SO_4 (1 + 1). To avoid loss of Cu the beaker in which the Cu is dissolved is covered with a clock-glass, placed in water and covered with an inverted beaker whose open end is below the surface of the water. When the reaction is complete the soln. is heated for 2 hr. on a steam-bath, combined with rinse water and the water from the Cu trap and diluted to 340 ml. The soln. is electrolysed between weighed electrodes at a current density of 1 amp per sq. dm. rising to 2 amp. The electrodes are removed, washed, dried and re-weighed. The electrolyte is evaporated to dryness, ignited and dissolved in water. The soln. is adjusted to pH 3·8 with acetic acid, treated with 1 ml of 1 per cent. aq. gum arabic, diluted to 100 ml and treated with 2 ml of 0·1 per cent. alcoholic rubeanic acid. After 10 min. the Cu content is determined colorimetrically and the figure found is added to that obtained by electrolysis. The error is $< \pm 0{\cdot}003$ per cent. D. Bailey

899. **Analytical chemistry of micro quantities of beryllium.** T. Y. Toribara and R. E. Sherman (*Anal. Chem.*, 1953, **25** [11], 1594–1597).—Colorimetric, fluorimetric and spectrographic methods of determining microgram quantities of Be, particularly in biological samples, are reviewed. A study of the completeness of each step in a separation scheme is reported. Organic matter is removed by evaporating an aq. soln. containing Be and igniting at up to 750° C. The salts are heated strongly with conc. H_2SO_4 and the Be is absorbed from soln. with a cation-exchange resin and extracted with acetylacetone. For small samples of urine (50 ml), tissues and filter-papers containing dust, a wet-ashing procedure with HNO_3 and H_2SO_4 or HNO_3 and perchloric acid is often used. For large quantities of urine, perchloric acid should not be used because of the large quantities of K present. A semi-dry technique with HNO_3 only is described. Most of the cations are removed by electrolysis and acetylacetone is used to complete the separation. By this scheme, recoveries from seven 500-ml samples of urine each containing 1·3 μg of Be and radio-isotope ^7Be ranged from 97·5 to 101·7 per cent. (average 99·9 per cent.). Two samples containing carrier-free radio-isotope ^7Be gave recoveries of 96·6 and 100·3 per cent. For bone, dry ashing at 600° C is most rapid. After most of the Ca has been removed as $CaSO_4$, the acid soln. is passed through a column of Dowex-50 in the acid form. If the bone contains more than 0·1 μg of Be per g of ash, the metal can be gathered with a calcium phosphate precipitate obtained by raising the pH of the soln. After soln. in H_2SO_4, the separation of the Be is completed by electrolysis at a mercury cathode and acetylacetone extraction. Recoveries of 95 to 98 per cent. have been attained for quantities of Be as small as the carrier-free radio-isotope ($< 10^{-10}$ g). O. M. Whitton

900. **The determination by radioactivation of the oxygen content of powdered metals with particular reference to beryllium.** R. G. Osmond and A. A. Smales (*Anal. Chim. Acta*, 1954, **10** [2], 117–128).—The O content of Be is determined by irradiation of a mixture of the powdered metal with $\simeq$ 7 times its weight of LiF. The reactions $^6Li(n,\alpha)^3H$ followed by $^{16}O(t,n)^{18}F$ take place and the ^{18}F ($\beta+$, half-life 112 $\pm$ 2 min.) is separated by distillation as H_2SiF_6, and, after separation of ^{38}Cl (^{38}Cl formed from impurity has a half-life of 38 min.), ^{18}F is pptd. and counted as PbClF. A self-absorption correction is made for the wt. of PbClF used and the experimental results are corrected to a wt. of 1 g. LiF usually contains some O, and, even after sintering "extra-pure" LiF, a "standard activity" of $\simeq$ 200 counts per min. is attained under the standardised conditions of 100 per cent. yield from 126 mg of LiF corrected for self-absorption at 240 min. (after 30 min. irradiation in the Harwell pile) with a counter geometry of 10·6 per cent. LiF is chosen,

as the formation of ^{18}F by (n,2n) reaction is negligible. Although Li has a high absorption cross-section it is shown that neutron self-shielding is insignificant. The optimum LiF to Be ratio for the irradiation is determined as > 4 to 1. The results of the method after standardisation with BeO or Be metal of known O content and with samples of Be containing $\simeq$ 1·2 or 0·3 per cent. of O are compared with unpublished results for the same samples by standard methods. All the experiments are carried out with Be of particle size $< 50\ \mu$, which is consistent with the range of tritium particles emitted from the Li. An appendix gives the detailed procedure for the determination.

E. J. H. BIRCH

901. **Heterometric micro-determination of magnesium with oxine in the presence of foreign metals.** M. Bobtelsky and Y. Welwart (*Anal. Chim. Acta*, 1954, **10** [2], 156–160).—A titration with NaOH of a soln. in aq. acetic acid of 0·01 *M* $MgCl_2$ (5 ml) and 0·02 *M* oxine (5 ml) shows an inflection between pH 7 and 9; pptn. is complete at pH 9. In the absence of other metals, 15 ml of an aq. soln. containing Mg ($\simeq$ 1 mg), *M* NH_4NO_3 (1 ml) and 4 *M* aq. NH_3 (5 ml) is titrated with 0·01 to 0·003 *M* oxine in 50 per cent. ethanol. When excess of Al, Zn, Cd, Mn or Ca are present sufficient sodium citrate (1 g) is added to complex the other metals before titration. The effect of the complexing agent itself is shown to be negligible. The height of the optical density max. is variable, but its position gives an accurate end-point if taken as the first point on the max. line. The error is small after some experience with the method. The complex is pptd. slowly so that the density readings must be made only when the galvanometer is steady and the soln. is continuously stirred.

E. J. H. BIRCH

902. **Uncombined calcium oxide or hydroxide in lime and silicate products. Volumetric determination.** G. O. Assarsson and J. M. Bokström (*Anal. Chem.*, 1953, **25** [12], 1844–1848).—Several methods for the volumetric determination of uncombined CaO and $Ca(OH)_2$ extracted from lime and silicate products were investigated. The solvents used for extraction were glycerol, ethylene glycol and acetoacetic ester, and the best results were obtained by conductimetric titration with a strong acid. Reasonable results were also obtained when indicators were used, methyl red being suitable for glycerol and ethylene glycol extracts and bromophenol blue for acetoacetic ester extracts. The only convenient method of titration, when an accelerator such as $Sr(NO_3)_2$ is used, is with an indicator.

G. P. COOK

903. **Indirect volumetric determination of zinc by separation as mercurithiocyanate.** F. Sierra and J. Hernández Cañavate (*An. Soc. Esp. Fís. Quím., B*, 1953, **49** [12], 773–776).—To a sample of $ZnSO_4$ or $Zn(NO_3)_2$ (0·05 to 0·3 g of Zn) in H_2O (10 or 20 ml) is added 10 per cent. aq. glycerol (10 ml) and then, at 100° C, 10 per cent. $Na_2Hg(CNS)_4$ (10 ml per 0·1 g of Zn). After leaving for 2 hr. to cool, the soln. is diluted to 50 ml and filtered through a dry paper. Half or quarter of the original vol. is titrated with 0·1 *N* $Hg(NO_3)_2$; *o*-dianisidine (1 per cent. in ethanol with 1 per cent. of acetic acid) and Fe alum is used as indicator. The titre is subtracted from the titre of a blank containing no Zn.

D. P. YOUNG

904. **Heterometric microtitration of aluminium with oxine.** M. Bobtelsky and Y. Welwart (*Anal. Chim. Acta*, 1954, **10** [2], 151–155).—On titrating Al with oxine there is a break in the curve of optical density plotted against added vol. only for the final product comprising 1 mol. of Al to 3 mol. of oxine. In the reverse titration sol. intermediates at ratios 1 to 2 and 1 to 1 are also indicated. Titration of an acid soln. equimol. with respect to Al and oxine with NaOH shows two points of inflection at pH 6·5 corresponding to pptn. and at pH 10·5 corresponding to solution of the ppt. Two methods for heterometric titration are given: in one, $> 0{\cdot}5$ mg of Al in 20 ml of aq. acetate buffer at pH 5·5 to 6 is titrated with 0·05 *M* oxine in ethanol, and in the other 1 to 2 ml of 5 *M* Na acetate is added to 20 ml of an aq. solution containing $> 0{\cdot}5$ mg of Al and the soln. is titrated with 0·02 to 0·05 *M* oxine in 0·1 to 0·25 *M* acetic acid. The end-point is at the first max. optical density point. The lower limit of Al concn. is 0·001 *M*. The error is $\pm$ 2 per cent.

E. J. H. BIRCH

905. **The titration of aluminium with complexone III at pH 3.5.** K. ter Haar and J. Bazen (*Anal. Chim. Acta*, 1954, **10** [1], 23–28).—Al is estimated at pH from 3·5 at room temp. to 4·3 at the b.p. by the addition of excess of complexone III soln. followed by back-titration with $Th(NO_3)_4$ soln. The results are 1 per cent. low, but are reproducible, so a practicable factor can be used. The cations Fe, Ni, Bi, Mn, Co, Cu, Pb, Zn and Cd interfere, the first three doing so quant. The anions F′, PO_4''', oxalate and SO_4'' also interfere, although the SO_4'' can be removed by addition of $BaCl_2$ soln. To 10 to 50 mg of Al in a vol. of $\simeq$ 100 ml, excess of 0·05 *M* complexone III soln. is added, and the soln. is neutralised to Congo red. Five ml of 2 *M* chloroacetic acid and 10 ml of *M* Na acetate are added, so that the pH is between 3·5 and 3·6. After the addition of 1·5 ml of 0·1 per cent. alizarin-S soln., the excess of the complexone is titrated with 0·05 *M* Th $(NO_3)_4$ soln. until the colour changes sharply from orange to red. For titration at pH 4·3, the buffering is achieved by 10 ml each of 2 *M* acetic acid and *M* Na acetate soln. The colour change is less sharp, going from a dark orange to red.

J. H. WATON

906. **Micro-titrations with ethylenediaminetetra-acetic acid. IX. Determination of indium. X. Direct titration of manganese in pure solutions and in the presence of other metals.** H. Flaschka and A. M. Amin (*Mikrochim. Acta*, 1953, [4], 410–413; 414–420).—**IX.**—In an ammoniacal soln. containing tartrate, indium forms a red complex with Eriochrome black T. The indium is removed from the complex with complexone III and the blue colour of the free dye appears at the end-point of the titration. The method works satisfactorily in the presence of Hg, Cu, Cd, Zn, Co or Ni, if these are masked by the addition of KCN. **X.**—In an ammoniacal soln. containing tartrate and reduced with ascorbic acid, Mn can be directly titrated with complexone III with Eriochrome black T as indicator. If Hg, Cu, Cd, Zn, Co or Ni are present they can be masked by the addition of KCN. Zn and Cd can be determined after the Mn titration by the addition of formaldehyde.

A. J. MEE

907. **Determination of impurities in germanium and silicon.** C. L. Luke and M. E. Campbell (*Anal. Chem.*, 1953, **25** [11], 1588–1593).—Quantitative

photometric methods of determining 0·1 to 1 p.p.m. of As, P, Sb and Cu in Ge and GeO_2 and a method for determining 1 to 10 p.p.m. of As in Si are described. The sample is dissolved in oxalic acid plus hydrogen peroxide, the As is isolated with diethylammonium diethyldithiocarbamate by chloroform extraction and determined by the molybdenum-blue method. To determine P, Sb and Cu, the Ge is first quantitatively removed by distillation as chloride from HCl - HNO_3 soln. containing a little perchloric acid. The molybdenum-blue method used for As is equally applicable to P. The Sb is determined by the photometric rhodamine B - benzene method, the Sb being oxidised to the quinquivalent state before addition of rhodamine B. In the photometric neocuproine method used for Cu, the coloured Cu compound is extracted with chloroform. Methods of preventing interference from metals are indicated.
O. M. WHITTON

908. **Quercetin as colorimetric reagent for determination of zirconium.** F. S. Grimaldi and C. E. White (*Anal. Chem.*, 1953, **25** [12], 1886–1890).—An intense yellow colour is obtained when quercetin is added to a soln. of Zr in 0·5 *N* HCl. The absorbancy is measured at 440 mμ at a slit width of 0·05 mm, Beer's law being obeyed with as much as 60 μg of ZrO_2. Interferences are many but they can be obviated by prior separation of the Zr with *p*-dimethylaminoazophenylarsonic acid (**I**). Only W, Ta, Nb, Sc and Th are pptd. by this reagent and interfere with the colorimetric finish. V and other strong oxidising agents should also be absent, and F′ hinders the pptn. of Zr with **I**. Under the conditions stated above it is indicated that quercetin forms a 2 to 1 complex with Zr although a 1 to 1 complex can co-exist under special conditions. Approximate values for the equilibrium constants of the complexes are: K_1 (2 to 1 complex) = 0·33 × 10^{-6} and K_2 (1 to 1) = 1·3 × 10^{-9}. Agreement was good with other methods, when standard samples of glass sands and refractories were analysed. A table giving the limiting concn. of various anions and cations in the colorimetric determination is included.
G. P. COOK

909. **Mandelic acid and halogen-substituted mandelic acids as reagents for the determination of zirconium.** R. Belcher, A. Sykes and J. C. Tatlow (*Anal. Chim. Acta*, 1954, **10** [1], 34–47).—The three fluoro- and the three trifluoromethylmandelic acids have been used as reagents for the pptn. of Zr, and further investigations have been made on the use of *p*-bromomandelic acid and of mandelic itself. Only the *p*-fluoro- and *p*-trifluoromethylmandelic acids are as sensitive as mandelic acid and its *p*-bromo derivative in the pptn. of Zr. The direct weighing of the ppt. with *p*-bromo-, *p*-fluoro- and *p*-trifluoromethylmandelic acids leads to low and variable results, but on ignition ZrO_2 is obtained quant. When mandelic acid is used, and the ppt. is washed with saturated Zr mandelate soln., results are consistent but low by direct weighing. Although an over-all empirical correction factor of 1·0145 can be used, greater accuracy is attained by use of a factor for the particular concn. range or by ignition to ZrO_2.
J. H. WATON

910. **Spectrographic estimation of lead in twig samples.** J. R. Butler (*Analyst*, 1954, **79**, 103–104).—The method described was devised for semi-quant. estimation of Pb in twigs located where sub-outcropping Pb-rich veins occur beneath soil. The twig, trimmed to a standard size, is fed into a vertical d. c. carbon arc near the anode, the upper region of the arc near the cathode being focused on the slit of a Hilger large quartz spectrograph. Much of the background radiation from the burning solid specimen is thus not recorded on the spectrogram. The intensities of the line Pb 2833·07 A are estimated against an arbitrary series of fixed intensities. With twigs of known Pb content it was found possible to relate the series of fixed intensities to the approx. Pb content. The limit of detection is ≃ 0·5 p.p.m. in the oven-dry material, and the reproducibility for line intensities corresponding to Pb contents up to 50 p.p.m. is ± 50 per cent. or better.
A. O. JONES

911. **A comparison of lead-isotope analysis techniques.** R. M. Farquhar, G. H. Palmer and K. L. Aitken (*Nature*, 1953, **172**, 860).—Isotopic lead analyses by different techniques and different mass spectrometers are compared. Good agreement is found between analyses made with tetramethyl lead and a 180° Nier-type spectrometer and those with solid lead compounds ($PbCl_2$ or PbI_2) and a 60° Nier-type instrument. The effects of mass discrimination appear to be negligible, although there is a slight discrepancy of ^{204}Pb.
D. BAILEY

912. **A contribution to the estimation of lead in steel.** H. A. Nicolas (*Chim. Anal.*, 1954, **36** [1], 8).—The method of Schoch and Brown is modified to make it applicable to the determination of lead in steel. A sample of the steel (2 g) is treated with 30 ml of conc. HCl in a 250-ml covered Erlenmeyer flask. The soln. is diluted with 50 ml of hot water, filtered through a rapid filter into a 300-ml electrolytic beaker and made up to about 200 ml with water. $NH_2OH.HCl$ (2 g) is added, and the soln. is electrolysed for 10 min. by a current of 3 amp. (rotating anode). Deposits up to 100 mg adhere satisfactorily; if the deposit is less than 5 mg, the wt. of sample is doubled. Recoveries are good with known solutions containing 5 to 50 mg of Pb per 2 g of sample.
E. HAYES

913. **Micro-titrations with ethylenediaminetetra-acetic acid. VIII. Estimation of thorium and aluminium.** H. Flaschka, K. ter Haar and J. Bazen (*Mikrochim. Acta*, 1953, [4], 345–348).—The method depends on the formation of complexes between complexone III and weekly acid solutions of Th and Al. An excess of 0·01 *M* complexone III soln. is added, the mixture is diluted, buffered with K acid phthalate in a mixture of acetic acid and Na acetate, and boiled. The hot soln. is titrated with $Th(NO_3)_4$ (previously standardised under the same conditions against complexone III) with alizarin-S as indicator to a red colour, and then with complexone III to a yellow colour.
A. J. MEE

914. **The spectrophotometric determination of hydrazine in dilute solutions.** J. P. Riley (*Analyst*, 1954, **79**, 76–81).—The method described for determination of hydrazine at concn. as low as 10^{-5} *M* is a development of that of Kulberg *et al.* (*Brit. Abstr. C*, 1952, 149), the ethanolic soln. of picryl chloride being replaced by a soln. in $CHCl_3$. After reaction and addition of alcoholic K acetate soln. the optical density is measured spectrophotometrically at 494 mμ. Beer's law is obeyed at concn. up to 30 p.p.m. If the concn. is below 1 p.p.m. or if hydroxylamine is present, the optical density is measured at 530 mμ.
A. O. JONES

915. **Detection and estimation of hydroxylamine with 8-hydroxyquinoline.** W. Prodinger and O. Svoboda (*Mikrochim. Acta*, 1953, [4], 426–433).—The reaction between hydroxylamine and 8-hydroxyquinoline in alkaline soln. gives an indo-oxine, the colour of which varies with pH. By buffering with glycine - NaOH it is possible to use this reaction in a photometric method for determining hydroxylamine. The Beer - Lambert law holds only for certain optimum reaction times. For 2 to 10 μg of hydroxylamine this is 4 hr. For 20 to 100 μg it is 1 hr. The lower limit for reproducible results is 1 μg per litre, but the sensitivity of the method can be increased by enrichment of the indo-oxine formed. Na indo-oxine is acidified with alcoholic acetic acid and the free indo-oxine is extracted with chloroform. The upper limit, which is 100 μg per ml, is governed by the slight solubility of the Na indo-oxine. All metals other than alkali metals must be removed before the determination. The reaction begins at pH 10 and reaches a max. at pH 11·5. Ammonium salts interfere only in 1000-fold excess. The method is used to determine the hydroxylamine produced when *cyclo*hexanone oxime is hydrolysed.

A. J. Mee

916. **Analysis of sodium pyro- and tripolyphosphate mixtures by X-ray diffractometer using an internal standard.** A. J. Mabis and O. T. Quimby (*Anal. Chem.*, 1953, **25** [12], 1814–1818).—A method, based on X-ray diffraction with MgO as an internal standard, for the determination of mixtures of crystalline $Na_4P_2O_7$, anhydrous $Na_5P_3O_{10}$ in its two forms and $Na_5P_3O_{10}.6H_2O$ is described. Special methods of sample preparation eliminate errors caused by preferred crystalline orientation. The average deviation of the method is $\simeq$ 3 per cent., according to analyses based on mixtures of known composition, the greatest error being found in the determination of $Na_5P_3O_{10}.6H_2O$. G. P. Cook

917. **Colorimetric determination of [quinquivalent] vanadium and its separation from copper. Use of cupferron.** H. H. Willard, E. L. Martin and R. Feltham (*Anal. Chem.*, 1953, **25** [12], 1863–1865).—The V is separated from soln. by pptn. with cupferron at pH 1·0 and a temp. of 0° to 10° C. The ppt. is filtered and washed with 10 per cent. H_2SO_4 until the washings are free from cupferron, which interferes with the final colorimetric finish if present in the ppt. The vanadium - cupferron complex is dissolved in acetone and the soln. is set aside for 20 to 30 min., before the absorbance at 740 mμ is measured. Beer's law is obeyed over the range of concentrations determined, *viz.*, 0·0056 to 0·14 mg of V per ml. The effect of Cu is eliminated by chelation with the tetrasodium salt of ethylenediaminetetra-acetic acid, the Cu complex formed being stable to cupferron and remaining in solution while the V is pptd. The average error of the method is ± 2·5 per cent. when Cu is present. G. P. Cook

918. **Determination of arsenic in kermesite.** M. A. Morette (*Ann. Pharm. Franç.*, 1953, **11** [5], 361-364).—Since the prescribed (French) method for estimation of As in kermesite (Sb_2O_3 and Sb_2S_3) tends to leave As_2S_3 with pptd. S on the filter, another method is proposed. The kermes mineral (1 g) is heated slowly with 20 ml of dil. HNO_3 and placed in a water-bath for 2 hr., when, after addition of 2 ml of dil. HNO_3 and 20 ml of H_2O, the soln. is filtered and the As that has been oxidised to arsenate passes into the filtrate. The filtrate is evaporated to dryness, and the residue is heated to fuming with H_2SO_4, dissolved in HCl and reduced with hypophosphite reagent; As gives a yellow-brown coloration. The test gives a positive reaction with 0·1 mg of As per g of material (0·03 mg per day in the max. permissible dose of kermes mineral). The method is applicable to Sb_2S_3, but the first addition of HNO_3 is made with conc. acid and the mixture is slowly heated until the black particles have disappeared. E. J. H. Birch

919. **Spectrophotometric determination of arsenic and tungsten as mixed heteropoly acids.** D. K. Gullstrom and M. G. Mellon (*Anal. Chem.*, 1953, **25** [12], 1809–1813).—The method for As is based upon the formation of molybdovanadoarsenic acid and the yellow colour it gives under suitable conditions. The reagent is prepared by mixing a soln. containing 25 g of $Na_2MoO_4.H_2O$ with a neutral soln. containing 3·75 g of $NaVO_3$ and 50 ml of conc. HCl and by dilution to 250 ml. Five ml of this reagent are added to the soln. of As, the final vol. is made up to 50 ml and the absorption is measured at any wavelength between 380 and 460 mμ as no absorption max. occur. $Bi^{\cdots}$, Pb, Th, $Zr^{\cdots\cdots}$, Ag, Cr_2O_7'', Ni, MnO_4', S_2O_3'', CNS', GeO_3'', PO_4''', SiO_3'', WO_4'', VO_3', tartrate, citrate and borate interfere seriously. The concn. range is 1 to 30 p.p.m. and the results are reproducible and accurate for mixtures containing As as Paris green. The method for W is based upon the formation of tungstovanadophosphoric acid, the resulting colour being measured between 380 and 460 mμ. The optimum pH required is 1·8 and the ratio of the mixed vanadate - phosphate reagent is critical. This reagent is prepared by mixing a neutral soln. containing 0·8 g of $NaVO_3$ with 20 ml of syrupy H_3PO_4 and diluting to 100 ml. The concn. range of the method is 10 to 120 p.p.m. and the method is suitable for determination of W in steel. Bi, Pb, Th, $Zr^{\cdots\cdots}$, Ag, Cr_2O_7'', MnO_4', S_2O_3'', AsO_4''', GeO_3'', oxalate, citrate and oxidising agents interfere seriously. G. P. Cook

920. **Ultra-violet spectrophotometric determination of niobium in hydrochloric acid.** J. H. Kanzelmeyer and H. Freund (*Anal. Chem.*, 1953, **25** [12], 1807–1809).—The method is based upon the formation of a niobium chloride complex in conc. HCl and the measurement of its absorption peak at 281 mμ. The absorbance increased with increasing HCl concn. and hence the max. concn. of this reagent is essential for max. sensitivity and accuracy. $V^{\cdots\cdots}$, $Cr^{\cdots}$, $Pb^{\cdot\cdot}$, $Fe^{\cdots}$, $Cu^{\cdot\cdot}$, Mo and $Ti^{\cdots\cdot}$ interfere, but interference from $Fe^{\cdots}$ and $Cu^{\cdot\cdot}$ can be eliminated by reduction with $Sn^{\cdot\cdot}$. Beer's law is followed up to 10 μg of Nb per ml and the standard error of a mean absorbance of 0·522 is ± 0·003.

G. P. Cook

921. **Ultra-violet spectrophotometric determination of tantalum with pyrogallol.** J. I. Dinnin (*Anal. Chem.*, 1953, **25** [12], 1803–1807).—A 0·05 to 1·0-g sample of tantalum-bearing rock is fused with 10 g of $KHSO_4$; 50 ml of 5 per cent NH_4 oxalate are added to the cooled melt, and the mixture is stirred on a steam-bath. An aliquot containing $\simeq$ 1 mg of TaO_2 is added to 25 ml of 8 *N* HCl, 10 ml of pyrogallol soln. (200 g of pyrogallol, 100 ml of conc. HCl and 10 ml of 2 *M* $SnCl_2$ per litre) and sufficient ammonium oxalate to make its final concn. 0·125 g per 50 ml. The soln. is made up to 50 ml

with water and the absorption at 325 mμ is determined. Most interferences are additive in character and can be readily obviated, separations or major corrections being necessary when significant amounts of Mo, W, Sb or U are present. F′ bleaches the colour even when present in trace amounts. The results agree well with those obtained by gravimetric procedures. G. P. Cook

922. **The titration of bismuth with complexone III at pH 2.0 to 2.8.** K. ter Haar and J. Bazen (*Anal. Chim. Acta*, 1954, **10** [2], 108–112).—The formation of a 1 to 1 complex of Bi with complexone III [disodium dihydrogen ethylenediaminetetra-acetate] hydrolysed above pH 8 is shown by titration with NaOH. Bi may be determined by adding excess of complexone III, buffering to pH 2·8 (chloroacetic acid - sodium acetate) and titrating with $Th(NO_3)_4$ soln. with alizarin-S as indicator. The titration may also be carried out after adjusting to pH 2·0 (without buffering, as the buffer interferes with the Th titration). At pH 2·8 Fe and Ni react quant. and Al nearly so; Cu, Pb, Zn and Cd interfere, but Co, Mn, Mg, Ca and Ba do not. At pH 2·0 Fe interferes quant., Ni seriously and Cu slightly. E. J. H. Birch

923. **The volumetric determination of hydrogen sulphide and soluble sulphides.** Per Olof Bethge (*Anal. Chim. Acta*, 1954, **10** [2], 113–116).—The iodimetric method of Zimmermann (*Brit. Abstr. C*, 1950, 387), in which sulphide absorbed in Cd acetate - Na acetate is mixed with neutral $I' - NO_3'$, acidified and titrated with $Na_2S_2O_3$, is critically investigated. Owing to side-reactions results are high whenever the mixing of the sulphide and $I' - IO_3'$ takes place before acidification. The amount of error depends upon the rate of addition of the reagents. Addition of the reagents to the vessel in the order (*i*) HCl, (*ii*) KI - KIO_3 and (*iii*) Cd acetate soln. containing the sulphide, gives reproducible and accurate results. E. J. H. Birch

924. **Determination of sulphur in insoluble sulphides by reduction at a mercury cathode.** J. Bertetti (*Ann. Chim. Appl., Roma*, 1953, **43** [3], 167–172).—Sulphides are reduced at a mercury cathode, in the presence of H_2SO_4, the H_2S produced is trapped in Cd acetate soln. and the CdS formed is titrated with I, giving good agreement with gravimetric determinations for the S content of pyrites, chalcopyrite, galena, blende and cinnabar. R. C. Murray

925. **A new manometric method for the determination of sulphide ion in solution.** R. E. Press and K. A. Murray (*J.S. Afr. Chem. Inst.*, 1952, **5** [1], 31–43).—The method is based on the catalytic effect of sulphide ion on the reaction between azide and iodine, the nitrogen evolved giving a measure of the sulphide ion. The kinetics of the reaction are discussed and the relation between the extent of reaction and concn. of reagents is shown graphically. A high relative azide concn., addition of sufficient iodine to allow completion of reaction, and a pH of reaction mixture of about 6 are necessary. The apparatus functions similarly to one of Warburg type with, however, modifications in the mechanism for stirring the contents of the flask. Sulphide over a range of 0.5 to 0.01 p.p.m. can be estimated, with a standard deviation approximately 10 per cent. of sulphide concn. M. Tadman

926. **Vacuum fusion analysis. Apparatus and determination of oxygen in chromium.** W. S. Horton and J. Brady (*Anal. Chem.*, 1953, **25** [12], 1891–1898).—A vacuum fusion apparatus for the determination of O, N and H in non-ferrous metals is described, special reference being made to the determination of O in Cr. The blank rates compare well with those of other apparatus but the blank rate is higher in the presence of stop-cock grease. Results are satisfactory when an iron-alloy bath containing 25 per cent. of Sn is used and the operational temp. is $\simeq$ 1600° C, the recovery being 85 per cent. when the sample is first added and dropping to $\simeq$ 70 per cent. when the Cr concn. in the bath reaches 5 per cent. G. P. Cook

927. **Quinquivalent tungsten as a reducing agent in potentiometric titrations.** A. Riad Tourky, I. M. Issa and A. M. Amin (*Anal. Chim. Acta*, 1954, **10** [2], 168–177).—Solutions of W^V are prepared by electrolytic reduction of tungstate in 10 *N* HCl, and the blue soln. obtained are stored and used under CO_2. Standardisation with $K_2Cr_2O_7$ (followed potentiometrically) is shown to be fairly accurate in 8 *N* HCl at 25° C when there is > 20 mg of W^V in the soln. At lower acidities or higher temp. the results are unreliable. The stability of W^V soln. are investigated and although unstable in air or O, they keep for 14 days under CO_2. The reaction with O is shown to be of the first order. The formal redox potential of W^{VI}/W^V is found to fall from 0·247 V in 10·5 *N* HCl to 0·065 V in 7·0 *N* HCl and from 0·174 V in 6·7 *N* HCl - H_3PO_4 to 0·162 V in 5·5 *N* HCl - H_3PO_4, rising again on further decrease of acid concn. to 0·308 V at 0·5 *N*. In the determination of Fe^{III} the equilibrium is attained slowly and the best conditions are in 8 to 10 *N* HCl at 80° C. For lower acidity the presence of H_3PO_4 improves the accuracy. The blue colour of the W^V ion can serve as a visual indicator or CNS′ may be used. Cu^{++} can be titrated in 8 *N* HCl at 80° C when the reduced state is effectively $CuCl_2'$. Iodate can be titrated with W^V but in the presence of H_3PO_4 alone a potentiometric curve is given that rises at first and then slowly decreases to the end-point. The reduction is not quant. in 2*N* HCl. Titration of W^V with IO_3' in 5 *N* HCl alone or with H_3PO_4, or in saturated KCl leads exclusively to ICl and the end-point inflection is sharp. E. J. H. Birch

928. **Separation and determination of tungsten and molybdenum by means of calcium chloride.** A. de Sousa (*Anal. Chim. Acta*, 1954, **10** [1], 29–33).—W and Mo can be separated after pptn. as $CaWO_4$ and $CaMoO_4$ by the solubility of the latter in HCl. The W then remains as a ppt. of tungstic acid. One g of mineral is fused with 1 g of Na_2CO_3 and 6 g of Na_2O_2 in a platinum crucible. After cooling and leaching out the melt, the soln. is filtered and HCl is added to the filtrate to give a pH of 8. The liq. is evaporated to $\simeq$ 200 ml and 50 to 60 ml of glycerol are added to prevent any ppt. from sticking to the sides of the beaker. Saturated $CaCl_2$ soln. (20 to 25 ml) is added and the soln. is boiled for 2 to 3 min. The ppt. of $CaWO_4$ and $CaMoO_4$ is filtered, washed with warm 5 per cent. $CaCl_2$ soln. and washed into a beaker. The vol. of liquid is reduced to 100 ml, if necessary 100 ml of conc. HCl are added and the soln. is boiled with stirring. The residue of tungstic acid is filtered, washed with hot water, dried and calcined to WO_3 at 800° C. The filtrate and washings are evaporated to $\simeq$ 100 ml, a few drops of phenolphthalein are added and aq. NH_3

is run into the boiling soln. until the indicator turns red. The boiling is continued for 2 to 3 min. to complete the pptn. of $CaMoO_4$, which is filtered, washed 3 to 4 times with 5 per cent. $CaCl_2$ soln. and twice with distilled water. The ppt. is dried and calcined at 800° to 850° C. The method has been used successfully for W and Mo estimations in several Mo ores, the whole determination being capable of completion in a day. The method can be adapted to W and Mo estimations in alloy steels. J. H. WATON

929. **Rapid polarographic method for the analysis of tungsten - cobalt alloys.** I. A. Bucklow and T. P. Hoar (*Metallurgia*, 1953, **48**, 317–318).—W - Co alloys can be rapidly analysed by direct polarography of the soln. formed by dissolving them in 50 per cent. v/v aq. H_3PO_4. Conc. HCl is used as supporting agent for $W^{\cdots\cdots}$ and *M* aq. NH_3 and *M* NH_4Cl for the $Co^{\cdot\cdot}$. W. ASHWORTH

930. **Determination of small quantities of uranium by means of a photographic emulsion.** G. Faraone (*Ann. Chim. Appl., Roma*, 1953, **43**, [3], 184–189).—The number of α-particle tracks visible under a microscope on a developed Ilford C_2 (100 μ) plate, after a drop of the U-containing soln. has been left on it for a specified time, is a measure of the U concn. R. C. MURRAY

931. **The determination of halogens in complex platinates. Preliminary reduction with hydrazine.** W. Pugh (*J. Appl. Chem.*, 1954, **4** [1], 47–48).—When K_2PtCl_6 or $(NH_4)_2PtCl_6$ is treated with $AgNO_3$ in cold or boiling soln. the ppt. is yellow and contains only one-half (approx.) of the wt. of Ag calculated to be equiv. to the total Cl present. During the analysis of the chloroplatinate of dimethylketazine it was observed that the aq. soln. decomposes on boiling with pptn. of platinum black; this suggested that hydrazine might be used for destroying the complex chloroplatinate anion before estimation of halogen with $AgNO_3$. The results of a series of experiments with various compounds are presented. The following procedure gives accurate results. The sample is dissolved in 30 ml of water and heated to boiling, an excess of hydrazine hydrate (5 ml of a 3 per cent. soln. for 0·2 to 0·3 g samples) is added, and the soln. is made distinctly alkaline with NaOH or aq. NH_3. After boiling for a few min. to coagulate the Pt, the soln. is filtered, the ppt. is washed with water and finally with dil. HNO_3, and the halide is determined in the soln. by the modification of the Volhard method described by Cadwell *et al.* (*Ind. Eng. Chem., Anal. Ed.*, 1935, **7**, 38).
J. M. JACOBS

932. **Determination of elemental fluorine.** I. Sheft, H. H. Hyman and J. J. Katz (*Anal. Chem.*, 1953, **25** [12], 1877–1879).—Br dissolved in BrF_3 can be titrated with F at room temp., the reaction being quant. and the end-point being readily detected by discharge of the Br colour. In this procedure F can be determined to ± 2 per cent. by means of the apparatus and pressure measuring device described; with more sensitive equipment the error should be reducible to ≃ ± 0·1 per cent. The apparatus is made from Ni and the titration cell from moulded Kel-F (chlorotrifluoroethylene high polymer). Other applications of the method are to the determinations of Br or substances that yield Br with BrF_3; they include metals, oxides and halides other than F. G. P. COOK

933. **A system of galvanometric analysis and its application to the determination of chlorides and sulphur.** C. Bordoni (*Ann. Chim. Appl., Roma*, 1953, **43** [3], 160–166).—The potential of the cell system Hg (+ ve) - soln. of substance to be titrated - Pt is plotted against amount of pptg. titrant. The end-point is indicated by a rapid rise and fall of potential, as the effect of one ion in lowering the mercury potential is first eliminated, and then replaced by that of another. The method is applied to the titration of NaCl with $AgNO_3$, and of $CuSO_4$ with $K_4Fe(CN)_6$ R. C. MURRAY

934. **The absorptiometric determination of perchloric and chloric acids in the electrolyte of lead - acid secondary cells after reduction by titanous sulphate.** C. F. Forster (*Analyst*, 1954, **79**, 90–95).—The method, (*a*), devised for determination of $HClO_3$ and $HClO_4$ in the H_2SO_4 electrolyte of secondary cells depends upon reduction of the acids to HCl with nascent H by means of a Zn - Cu couple in presence of titanous sulphate, addition of $AgNO_3$ under conditions that prevent coagulation of the AgCl and measurement of the turbidity by means of a photo-electric absorptiometer. A separate determination with omission of the titanous sulphate is made for $HClO_3$ and HCl together, (*b*), and one with omission of both H and titanous sulphate for the HCl alone, (*c*), are also made. The difference between (*a*) and (*b*) gives a value for the $HClO_4$ and that between (*b*) and (*c*) one for the $HClO_3$. The method is valid for concn. of $HClO_4$ up to 15 p.p.m.; with greater concn., dilution is necessary.
A. O. JONES

935. **Concentration of traces of elementary iodine [in water] and its microchemical separation from ionised iodine.** H. Spitzy, H. Skrube and F. S. Sadek (*Mikrochim. Acta*, 1953, [4], 375–385.)—The method depends on the four-stage extraction of iodine from solutions containing it and iodide ions by CCl_4. The iodine in the extracts is converted into iodide ion by treatment with KOH. After concentrating the soln. its iodine content is determined volumetrically by the Winkler method. The method permits of the determination of 4 μg of iodine in the presence of 10,000 times as much iodide. The method has been applied to the determination of free iodine in natural spring waters. A. J. MEE

936. **Amperometric titration and voltammetric determination of iodide with rotated platinum-wire indicator electrode.** I. M. Kolthoff and J. Jordan (*Anal. Chem.*, 1953, **25** [12], 1833–1837).—I' is determined voltammetrically by measurement of its anodic diffusion current in *M* H_2SO_4 at a potential of + 0·65 V. The range of concentration is 10^{-6} to 10^{-3} *M* and the precision is ± 1 per cent. for a 10^{-4} *M* soln. of I'. Br' does not interfere; Cl' gives high and erratic results, but its interference can be eliminated by adding HCN to the supporting electrolyte. I' also gives a well-defined anodic wave in *M* H_2SO_4 containing 20 per cent. of acetone, over the same range of potentials as in the presence of HCN, *viz.*, from + 0·65 to 0·95 V. I' is also determined by amperometric titration with standard KIO_3 or $KMnO_4$ in H_2SO_4 soln. to an I' or ICN end-point at + 0·65 V *vs* the S.C.E. In the absence of Br' and at Cl' concn. < 0·1 *M* and in the presence of 0·01 *M* HCN, I' can be titrated with either $KMnO_4$ or with Ce^{IV} at a potential of + 0·2 V. Another method involves the conversion of I' to IO_3' by use of Cl water and

amperometric determination by (*a*) titration with standard KI soln. in *M* H_2SO_4 at 0·65 V or (*b*) reduction of IO_3' to I′ by addition of excess of KI, the liberated I being titrated with standard $Na_2S_2O_3$ at + 0·2 V. The amperometric methods give a precision and accuracy of ± 0·2 per cent. in the 10^{-5} to 10^{-3} *M* concn. range and of ± 1 to 5 per cent. at concentrations as low as 10^{-7} to 10^{-6} *M*.

G. P. Cook

937. **Reaction of [ferrous] iron with *cyclo*hexane-1:2-dionedioxime in acid solution.** C. V. Banks and E. K. Byrd (*Anal. Chim. Acta*, 1954, **10** [2], 129–138).—The reaction of Fe^{II} with *cyclo*hexane-1:2-dionedioxime (nioxime) at low pH and in acetate buffer is studied with regard to Fe interference in Ni determinations. In 0·1 *N* acid solutions the absorption spectrum of nioxime varies irreversibly with time, forming an unidentified reaction product. Acetate at pH > 3 with nioxime gives the sum of the individual spectra. The complex forms slowly, equilibrium not being attained in 3 weeks, so that Fe^{II} solutions must be stored under N. The absorption spectra of Fe^{II} and Fe^{III} complexes are measured at various pH, and from the shape of the absorption curves for Fe^{III} and its time of formation it is assumed that the complexes are identical, the Fe^{III} being reduced to Fe^{II}. This is confirmed by the discharge of the colour with oxidising agents which do not destroy the nioxime. The plot of absorption against composition (Job's method) shows that 2 mol. of nioxime are associated with 1 atom of Fe in the complex. The complex is prepared as a black finely divided solid by pptn.; it decomposes at 105° C and has 2 mol. of nioxime and 2 mol. of H_2O to each Fe atom. E. J. H. Birch

938. **Electrolytic deposition of iron as an analytical technique.** J. O. Lay (*Metallurgia*, 1953, **48**, 313–314).—The electrolysis of Fe in oxalate soln. was investigated, complete deposition being readily achieved. Related elements were co-deposited, however, and it was found impossible to prevent this. A tentative method for Fe in blast-furnace slags was devised. A 0·2-g sample of slag was moistened with H_2O and decomposed with 5 ml of HCl (sp. gr. 1·16) and a few drops of HNO_3 (sp. gr. 1·42). Twenty ml of dil. H_2SO_4 (1 + 3) were added, the soln. was evaporated and fumed over a flame. The extract was cooled, the sides of the beaker were washed down and the extract was re-fumed. The extract was diluted to ≃ 25 ml and boiled, 100 ml of 5 per cent. NH_4 oxalate were added and the soln. was gently evaporated to ≃ 75 ml. After cooling to ≃ 50° C the soln. was filtered through a close-texture paper or pad and washed with water to give a final vol. of ≃ 120 ml. Five ml of oxalic acid soln. (10 per cent.) were added with a further 5 ml after 15 min., and the soln. was electrolysed with a platinum-gauze cathode and rotating spiral anode for 30 min. at 6 V and 14 amp. The sample should be adjusted to give 0·01 to 0·04 g of Fe. A little HF may be added to assist decomposition. The pH should be ≃ 4 at the beginning of electrolysis. In presence of Mn, 2·5 g of ammonium persulphate should be added and the soln. boiled and cooled before addition of oxalic acid. W. Ashworth

939. **The quantitative analysis of iron-stones containing small amounts of titanium, vanadium, manganese, chromium and phosphorus.** D. N. Grindley, E. H. W. J. Burden and A. H. Zaki (*Analyst*, 1954, **79**, 95–100).—A routine method is described. The ore is brought into soln. by treatment with HCl, fusion of residual insol. material with $KHSO_4$ and re-fusion of insol. matter still remaining with fusion mixture. SiO_2 is determined in the soln. in the usual way, Fe is extracted from the strongly acidified soln. with ether, and Ti, Va and Mn are determined colorimetrically, Cr volumetrically and P gravimetrically in aliquot portions of the Fe-free soln. A. O. Jones

940. **Recommended method for the spectrographic analysis of low-alloy steels.** British Standards Institution (B.S. 1121B: 1953, 21 pp.).—The samples are excited by means of a simple condensed spark discharge (controlled source units have not been found to improve the reproducibility). A flat sparking surface technique is used, the emitted spectral radiations being dispersed by a large quartz prism spectrograph and recorded on a photographic plate. The internal standard method is used to make an evaluation from the spectrogram produced. Densities are compared by means of a non-recording densitometer and the percentage concentration of the element is obtained from comparison with standard analysed samples. Indications are given in the text of when, if necessary, deviations can be made from the standard procedure without impairing the reproducibility. The method is applicable to the analysis of low-alloy steels of iron content 95 ± 1 per cent.; compositions outside this range need a correction. The elements determinable and their concentration ranges are as follows: silicon, 0·05 to 0·80 per cent.; manganese, 0·05 to 1·50 per cent.; nickel, 0·10 to 5·00 per cent.; chromium, 0·05 to 3·00 per cent.; molybdenum, 0·05 to 1·50 per cent.; vanadium, 0·03 to 0·65 per cent; and copper, 0·05 to 0·50 per cent.

The sample can vary considerably in form and the only preparation necessary is a clean, flat surface such as can be produced by manual filing. The metallurgical condition of the sample does not affect the analysis unless the sample is distinctly heterogeneous. Recommended line pairs for the above mentioned elements, standard iron lines and a spectrum chart are also included.

C. J. Keattch

941. **Rapid photometric determination of cobalt in the presence of iron.** J. N. Pascual, W. H. Shipman and W. Simon (*Anal. Chem.*, 1953, **25** [12], 1830–1832).—A neutral sample is boiled with 2 or 3 drops of Br until no more Br fumes are evolved, 2 ml of 33 per cent Na acetate are added and then 33 per cent. KF soln. is added dropwise until the amber colour of ferric acetate disappears. The soln. is then treated with 2 or 3 drops of the KF soln., 1 or 2 drops of conc. HCl and 1 ml of 0·5 per cent. nitroso-R salt. The soln. is boiled and 1 ml of conc. HNO_3 is added and any solids present are removed by filtration or centrifugation. The sample is cooled and diluted and its absorption is measured at 525 mμ against a blank prepared in the same manner. Cu^{++} interferes at concn. greater than 16 μg per ml and MnO_4' at concn. greater than 36 μg per ml. As much as 120 μg of Fe^{+++} per ml does not interfere. The standard deviation is ± 2 per cent. for 20 μg of Co and is less than ± 1 per cent. for 30 μg or more. G. P. Cook

942. **Quantitative inorganic paper chromatography. Direct determination of nickel in microgram quantities.** S. V. Vaeck (*Anal. Chim. Acta*, 1954, **10** [1], 48–67).—When a drop of a soln.

containing Ni in 3 *N* HCl is developed with a mixture of 90 ml of acetone, 10 ml of 25 per cent. HCl and 2 ml of acetylacetone, the impurities are carried away whilst the Ni remains more or less at the starting position. The Ni is revealed with rubeanic acid, the spot is cut out and the Ni is estimated spectrophotometrically. The sample containing Ni is dissolved and the vol. of the soln. is adjusted so that 100 ml contains from 1 to 20 mg of Ni in 3 *N* HCl. Part of this soln. is diluted by a known amount of water, say to half the Ni concn., so that the Ni is in soln. at 2 different strengths. Six points are marked on a 23 × 23-cm sheet of Whatman No. 1 filter-paper, 5 cm from one end. Then 0·045 ± 0·005-ml drops of both soln. and of 3 different standard Ni soln. are spotted on the paper at the points, the sixth position being used as a blank. The whole analysis is carried out in duplicate. The sheet is dried at room temp. at 50 to 60 per cent. humidity for ≃ 45 min., tied in the form of cylinder, and then developed by ascending chromatography for ≃ 90 min. The paper is then dried for 30 min. at room temp. and for 30 min. at 50° to 60° C. Residual acidity is neutralised with NH_3 vapour, and the paper is sprayed with a 0·05 per cent. rubeanic acid soln. in alcohol containing 5 per cent. v/v conc. aq. NH_3. The blue Ni spots and the blank are cut out as circles 27 mm in diameter, and their reflectances are measured in a Beckman DU spectrophotometer at 625 mμ. If large amounts of Al, Cr, V or Ti are present, a preliminary extraction of the Ni - dimethylglyoxime complex in $CHCl_3$ is necessary. The complex is dissolved by extraction with 10 ml of 3 *N* HCl. Good results are found for the Ni content of standard N -, Ni - Cr -, C - and high-speed steels, as well as of standard Mn brass and Al alloys. J. H. WATON

943. **The molecular and ionic solubility of nickel dimethylglyoximate.** H. Christopherson and E. B. Sandell (*Anal. Chim. Acta*, 1954, **10** [1], 1–9).—Solubility determinations in alkaline soln. give a value of 0·632 g per litre for the solubility of dimethylglyoxime (H_2Dx) in water at 25° C for an ionic strength of 0·05 *M* with a dissociation constant of $2{\cdot}6 \times 10^{-11}$. Also at ionic strength 0·05 *M* and at the same temp., the Ni - dimethylglyoxime complex has a mol. solubility corresponding to 57 ± 3 μg of Ni per litre, a solubility product of $4{\cdot}3 \times 10^{-24}$ and a dissociation constant of $4{\cdot}4 \times 10^{-18}$. The constancy of the values of the mol. solubility and solubility product show that over a pH range 2 to 9 the solubility is accounted for by $Ni^{\cdot\cdot}$ and $Ni(HDx)_2$, so that there is no significant contribution from other ions in soln. such as $(NiHDx)^{\cdot}$. From the values for the mol. solubility of $Ni(HDx)_2$ in $CHCl_3$ and water saturated with $CHCl_3$ at ionic strength 0·05 *M* and from the values for the two dissociation constants, the extraction coeff. for $Ni(HDx)_2$ between $CHCl_3$ and H_2O as given by the expression—

$$\frac{[Ni(HDx)_2]_{CHCl_3}\,(aH^{\cdot})^2}{[Ni^{\cdot\cdot}]\;[H_2Dx]^2_{H_2O}}$$

is 0·063 at 25° C. On pptg. Ni as $Ni(HDx)_2$, loss due to ionic solubility can be made negligible by using an excess of reagent, but there is still a loss owing to mol. solubility. For a 1-g sample in a vol. of 300 ml, this loss would amount to ≃ 0·002 per cent. of Ni. J. H. WATON

944. **Determination of ash in coke of low ash content.** A. Grossman and D. Marie (*Przem. Chem.*, 1953, **32** [8], 407–409).—Special care must be taken in the disintegration of low-ash coke for analytical purposes and in the preparation of test samples in order to avoid contamination with foreign matter from mills, mortars, etc. The coke is crushed in a hard-steel mill to 0·5 mm grains, then a 10-g sample is ground in an agate mortar to powder size (< 0·25 mm); finally any Fe particles present are removed with a magnet. The results are accurate enough to assess whether a coke is suitable for the production of carbon electrodes, etc. It has also been established that ash obtained by incineration of coke has much the same chemical composition as the ash from the coal used to produce the coke in question. H. BURSTIN

945. **Comparison of spectrochemical and semi-micro-methods in analysis of petroleum ashes.** G. V. Dyroff, J. Hansen and C. R. Hodgkins (*Anal. Chem.*, 1953, **25** [12], 1898–1905).—Samples of ashes from various petroleum fractions were analysed by spectrochemical and semi-micro-procedures in order that the results might be evaluated statistically. The results of this study showed that the precision and accuracy of the two methods are comparable. G. P. COOK

See also Abstracts 881, 886, 960.

3. ORGANIC ANALYSIS

946. **The interpretation of organic chemical analyses with anticomposition tables.** H. H. Hatt (*Chem. & Ind.*, 1954, [2], 30–32).—All possible empirical formulae corresponding with a given analysis are readily deduced if, instead of presenting the data in order of increasing mol. complexity, they are arranged in order of increasing percentage of carbon content, then, when this is const., in order of increasing H content, and so on. A sheet of such tables is shown for compounds containing C, H and O over the range C 83·77 and H 10·36 to C 84·39 and H 9·69 per cent. H. WREN

947. **Micro-determination of active hydrogen in organic compounds by lithium aluminium hydride.** D. Subba Rao, G. D. Shah and V. S. Pansare (*Mikrochim. Acta*, 1954, [1], 81–84).—Lithium aluminium hydride can be used advantageously for the determination of active hydrogen in organic compounds. It reacts more vigorously than the Grignard reagent ($MgCH_3I$), thus reducing the extent of competitive side reactions and steric effects. It is also soluble in a number of inert solvents. A simple Roth apparatus with some modifications in procedure to achieve accuracy and simplicity was used. In the method used no blank need be carried out as the solid lithium aluminium hydride is allowed to react with traces of moisture in the solvent and a volume of gas equal to that of the hydrogen evolved is allowed to escape before the solution is mixed with the substance to be analysed. A. J. MEE

948. **Micro-analytical determination of carbon and hydrogen.** F. R. Cropper (*Mikrochim. Acta*, 1954, [1], 25–48).—The quality of PbO_2 used for the absorption of oxides of N in a carbon - hydrogen combustion train can vary greatly. A practical test for measuring the efficiency and relative capacity of batches of PbO_2 for the absorption of NO_2 is described. Some batches have a short life because of low relative capacity; others with a high relative capacity give erratic values for C. The relative capacity is dependent on primary particle

size of the PbO_2 powder. Granules of suitable capacity can be made from PbO_2 powder of low capacity by ball-milling; alternatively the substance may be prepared by the action of NaOCl on Pb acetate. The erratic results for C are discussed; it is probable that CO_2 is held on absorptive spots on the PbO_2 surface. Retained CO_2 can be displaced by NO_2 in a subsequent test. A. J. MEE

949. **Use of the Garner balance in the investigation of errors in the carbon - hydrogen determination.** J. A. Kuck, P. L. Altieri and A. K. Towne (*Mikrochim. Acta*, 1954, [1], 1–16).—The reproducibility of weighing small-size glass Ascarite and Drierite absorption tubes with 0·25-mm capillaries on the Garner balance has been investigated. A "standard error of estimate" for an Ascarite tube was found to be ± 1·65 μg and for Drierite ± 2·65 μg. The results for aluminium absorption tubes were rather higher. Filled glass absorption tubes showed a steady increase in weight when standing in air. A freshly-filled Ascarite tube does not absorb moisture at a regular rate. Absorption tubes filled with oxygen undergo a loss in weight, particularly over the first two hours. Experiments with tubes of different capillary diameters show that the diameter of the capillary constriction is a most important critical dimension whenever precise milligram or decimilligram analyses are involved. Where the diameter is greater than 0·2 mm there is justification for deducting a correction based on time lapse before weighing. A. J. MEE

950. **Dumas nitrogen determination with decimilligram samples.** J. A. Kuck and P. L. Altieri (*Mikrochim. Acta*, 1954, [1], 17–24).—A small Dumas nitrometer of 0·2-ml capacity graduated in microlitres, and permitting estimation of tenths of microlitres is used, and its construction is described. Samples between 0·1 and 0·4 mg are weighed on a Garner quartz-fibre balance. The error for nitrogen over 10 analyses with 0·4-mg samples of azobenzene was ± 0·28 per cent. For 0·1-mg samples the error was ± 0·96 per cent. A. J. MEE

951. **Micro-analytical determination of sulphur and chlorine or bromine by means of magnesium. I.** W. Schöniger (*Mikrochim. Acta*, 1954, [1], 74–80).—The sulphur and halogen content of organic substances can be determined microchemically by decomposition with Mg. Only one sample is required. The compound is ignited with magnesium in a small tube. The S content is then determined iodimetrically by the Zimmermann distillation method, and the halogen is determined argentimetrically by the Kainz and Resch modification of the Volhard method. It is also possible to determine nitrogen by a similar procedure. A. J. MEE

952. **Micro-determination of primary amino-groups in aliphatic and aromatic compounds, amino-acids and peptides, carboxylic amides and sulphonamides.** G. Kainz (*Mikrochim. Acta*, 1953, [4], 349–365).—The action of nitrous acid on all primary amino-groups can be used to determine quantitatively all types of compound containing this group, no matter how bound, if the reaction is carried out not only in the cold (20° C) but also at higher temp. (100° C). On heating, the reaction time is reduced to 20 to 40 min.; in the cold the period is variable and may be as much as 8 hr. A simple apparatus is described which enables the reaction to be followed directly in the nitrometer. The number of amino-groups in sparingly-soluble peptides can be determined successfully by this method. Many anomalies in the method previously used by Van Slyke are shown to be due to the presence of a primary *iso*nitroso group or to an intermediate *iso*nitroso compound arising from active methylene groups. All these anomalies can be avoided by adding iodide to the de-aminating soln. A. J. MEE

953. **Micro-determination of carbonyl groups with nitro-substituted phenylhydrazines.** W. Schöniger, H. Lieb and K. Gassner (*Mikrochim. Acta*, 1953, [4], 434–446).—The sample is condensed with 2 : 4-dinitro-, 4-nitro-, or 2 : 4 : 6-trinitrophenylhydrazine. The liquid is filtered and the excess of the substituted phenylhydrazine is reduced with $TiCl_3$, the excess of $TiCl_3$ being determined with ferric ammonium sulphate. The quant. reaction between substituted phenylhydrazines and carbonyl compounds is not general, but if a sample of a crystalline phenylhydrazone can be isolated it is possible to determine the number of carbonyl groups in the original compound by a procedure similar to the above. A. J. MEE

954. **A new method of separation of organic compounds.** R. C. Vasisth and M. S. Muthana (*Nature*, 1953, **172**, 862–863).—A method of separating organic compounds by fractional crystallisation on filter-paper is described. The mixture in a volatile solvent is deposited on a filter-paper strip by suspending the strip with its lower end in the soln., which is then allowed to evaporate. The strip is then irrigated with a pure solvent or a mixture of solvents to separate the components of the mixture. The process is repeated to give pure fractions. Four mixtures separated by this method are: phenanthrene and anthracene; naphthalene and diphenyl; 1- and 2-naphthol; and hydroquinone and resorcinol. D. BAILEY

955. **Gas chromatography. I. The separation and estimation of volatile organic compounds by gas - liquid partition chromatography.** N. H. Ray (*J. Appl. Chem*, 1954, **4** [1], 21–25).—The method developed by James and Martin (*Brit. Abstr. C*, 1952, 199) for the separation and estimation of volatile fatty acids has been extended to the separation of a wide range of org. compounds, including hydrocarbons, alcohols, aldehydes and ketones, on a column of kieselguhr impregnated with di-(3 : 5 : 5-trimethylhexyl)phthalate. The outlet of the column was maintained at slightly reduced pressure to facilitate determination of the components by means of a thermal-conductivity cell. With the column at 110° C and the cell at room temp. and 75 mm pressure, substances with b.p. up to 180° C could be detected and estimated. The apparatus and the procedure are described and the results obtained in the separation of typical mixtures are presented. As an indication of the sensitivity of the method, it was found that 50 μl of a 0·01 per cent. solution of ether in benzene injected for the separation of aromatic hydrocarbons gave a measurable peak (≃ 2 chart divisions) for ether. J. M. JACOBS

956. **Titration of acids and bases in non-aqueous solution. II.** H. Ballczo (*Mitt. Chem. ForschInst. Öst.*, 1953, **7** [6], 126–131).—Alkyl- and arylamines, amino-acids, amino-alcohols, cyclic nitrogen bases, alkylene-oxides, sulphonamides, vitamins, alkaloids and their salts with organic and inorganic acids

can be titrated potentiometrically in glacial acetic acid, conductimetrically in thionyl chloride and by means of indicators. A description of the procedures for macro- and micro-titrations is given. W. GOOD

957. **Some aspects of chromatographic analysis in organic chemistry.** M. Servigne (*Chim. Anal.*, 1954, **36** [1], 3–8).—A discussion of the factors influencing the chromatographic separation of organic compounds is based on experiments in the separation of vitamin A from complex mixtures. A resumé is given of the treatment developed in the publication "*Fractionnement chromatographique et dosage de la vitamine A*" by M. Servigne, P. Guérin de Montgareuil and M. Pinta (Editions du Centre National de la Recherches Scientifique). Elution curves for a soln. containing a single constituent and for soln. containing several constituents are studied. E. HAYES

958. **The iodoform reaction.** C. J. de Wolff (*Pharm. Weekbl.*, 1954, **89** [3–4], 40–43).—A mixture of equal vol. (1 to 5 μl of each) of the test soln. and alkaline I in KI soln. are heated for 5 to 10 min. to 100° to 110° C in a sealed capillary tube 5 cm long of 1 mm bore; $\simeq$ 1 cm of the empty end of the tube projects free of the heating surface (metal block). In positive tests, crystals of CHI_3 sublime into the cooler part of the tube, and can be observed microscopically. The sensitivity is 40 p.p.m. for ethanol and 3 p.p.m. for acetone. From observations on a number of compounds, it is concluded that the reaction is probably given by all compounds (in addition to the well-known examples) containing the groupings $CH_3{\cdot}CH_2{\cdot}NR_1R_2$ or $CH_3{\cdot}CH(CH_3){\cdot}NR_1R_2$, where R_1 or R_2 are alkyl or aryl groups or H. The positive reactions obtained with procaine, ephedrine, and alkyl- or arylamines, and the negative results obtained with butacaine and amethocaine are thus explained. P. S. ARUP

959. **Co-operative mass spectrometric analysis of C_1 to C_4 hydrocarbon mixtures.** J. Blears and J. D. Waldron (*J. Inst. Petrol.*, 1954, **40**, 1–6).—The accuracy of British-manufactured sector-type mass spectrometers in hydrocarbon analysis is checked by analysing 4 synthetic C_1 to C_4 paraffin - olefine mixtures severally on each of 8 instruments. Computation of the results on the assumption that only the blended components are present in the mixtures, gives the average difference per component between the calculated and blended composition on the average instrument as 0·40 mole per cent. Much of this error arises through the collective effect of small random errors in cracking patterns, relative sensitivities and in measuring the peak height, but some may also result from genuine systematic errors in the instruments concerned. A general method of computation introduces slightly larger differences and also shows the presence of small amounts of gases considered to be absent from the blends. The results are compared with those of a similar co-operative analysis programme reported by Starr and Lane (*Brit. Abstr. C*, 1949, 384). D. R. GLASSON

960. **Continuous colorimetric determination of small amounts of oxygen in ethylene.** M. Struszynski, J. Minczewski, S. Waszak and J. Waclawik (*Przem. Chem.*, 1953, **32** [9], 449–457).—L. J. Brady's method (*Brit. Abstr. C*, 1949, 203), which is based on the change of colour of an alkaline soln. of reduced sodium anthraquinone-β-sulphonate by oxygen, has been modified and adapted for continuous recording of oxygen contents from 0·002 to 0·02 per cent. in C_2H_4, with an absolute error of $\pm$ 0·0005 per cent. The range of measurements can be extended to 0·1 per cent. by adjusting concn. of the reagent, diameter of capillary and flow of C_2H_4. The prep. of reagents, assembly and calibration of the apparatus and the procedure are described in detail. Diagrammatic sketches, calibration curves and a survey of literature are presented. H. BURSTIN

961. **Sunbury X-ray absorption method for the rapid determination of sulphur in hydrocarbons.** R. W. Cranston, F. W. H. Matthews and N. Evans (*J. Inst. Petrol.*, 1954, **40**, 55–63).—A simple, speedy and sufficiently accurate method is described for the determination of S by means of X-ray absorption for use in the control of refinery processes. Details are given of the principle of the method, the equipment required and the procedures for calibration and operation of the apparatus. The average times for a single determination and the accuracies attainable are: for distillate samples, about 5 min. and $\pm$ 0·03 per cent. S; and for residual samples, 5 to 15 min. and $\pm$ 0·1 per cent. S. Experimental results are tabulated to show the reproducibility of the method, and the results obtained are compared with those by conventional methods. Other possible applications are mentioned. G. C. JONES

962. **The separation and identification of normal aliphatic alcohols.** H. van Duin (*Rec. Trav. Chim. Pays-Bas*, 1954, **73** [1], 68–77).—Quant. separation, of mixtures of primary and secondary *n*-aliphatic alcohols by partition chromatography *via* their 4-dimethyl- or 4-diethylamino-3:5-dinitrobenzoic acid esters is described; silica gel and nitromethane as the immobile and light petroleum as the mobile phase are used. Homologues to C_{10} or C_{12} are completely separated and C_{10} or C_{12} to C_{18} when they differ by two C atoms; they are identified by their true retention vol. (see Abstract 968 below) and quantitatively estimated by measurement of ultra-violet absorption maxima density. Theoretical reasons are deduced for the linear relationship between the logarithm of the true retention vol. and the number of C atoms in the homologue, assuming ideal behaviour, the slope of the line being determined by the particular system and the homologous series. D. E. BLENFORD

963. **Chromatographic separation of glycols and monohydric alcohols.** S. Dal Nogare (*Anal. Chem.*, 1953, **25** [12], 1874–1877).—The monohydric alcohol separations are performed on a silicic acid column with water as the immobile phase; various mixtures of CCl_4 and $CHCl_3$ are used for resolution of the alcohols. The alcohol content of each fraction is determined by dichromate oxidation, excess of dichromate being determined by iodimetric titration. Glycol mixtures are resolved on a Celite - silicic acid column with water as the immobile phase and mixtures of *n*-butanol and $CHCl_3$ as mobile phases. The glycol contents of the fractions are determined by reaction with $NaIO_4$, the excess being determined by titration with NaOH to alizarin yellow R indicator. The separations are well defined and recoveries of 90 to 110 per cent. are attained. G. P. COOK

964. **Glycols and related compounds. Determination of propylene glycol in medicinal mixtures.** H. Isacoff (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 734–736).—Recoveries in collaborative determinations (*J. Ass. Off. Agric. Chem.*, 1952, **35**, 579) of propylene glycol in presence of glycerol and other substances were in the range of 95·7 to 110·9 per cent. A. A. ELDRIDGE

965. **Dichromate oxidation of diethylene glycol.** M. J. Cardone and J. W. Compton (*Anal. Chem.*, 1953, **25** [12], 1869–1874).—$K_2Cr_2O_7$ oxidation of diethylene glycol in 50, 25 and 12·5 per cent. H_2SO_4 at 100° C proceeds in a stoicheiometric manner to the oxidation levels represented by the oxidation numbers 20·0, 16·0 and 14·0, respectively. The oxidations are independent of the glycol and $K_2Cr_2O_7$ concn., provided that there is an excess of $K_2Cr_2O_7$. Application to the differential oxidation method for the resolution of a diethylene glycol and ethylene glycol mixture is accurate to within ± 5 per cent. of the glycol content. G. P. COOK

966. **Paper chromatography of some carbohydrates and related compounds in the presence of boric acid.** G. R. Barker and D. C. C. Smith (*Chem. & Ind.*, 1954, [1], 19–20).—The effect on the R_F values of various methylated sugars produced by running chromatograms on paper impregnated with H_3BO_3 and developing with a mixture of *n*-butanol saturated with HBO_2 and saturated aq. H_3BO_3 is reported. In presence of H_3BO_3 certain mixtures of sugars, which are otherwise not resolved, can be separated. Useful separations of sugar alcohols can also be achieved by the use of paper chromatography in presence of H_3BO_3, but, on the whole, the results with unsubstituted sugars are less promising than with methylated sugars. J. M. JACOBS

967. **Determination of glucose by means of sodium chlorite.** H. F. Launer, W. K. Wilson and J. H. Flynn (*J. Res. Nat. Bur. Stand.*, 1953, **51** [5], 237–245).—The kinetics of the spontaneous decomposition of ClO_2' in acid buffer solutions (second order) and of the oxidation of aldoses (glucose and cellobiose) with ClO_2' (rate α [aldose] × [ClO_2']) are studied, and the ClO_2' consumed by the aldose is given by the difference between ClO_2' consumed in a test and control soln. For practical determinations, a calibration curve (not linear) is made by plotting ClO_2' consumed after 20 hr. by the aldose at 40° C, at pH 3·52 and in presence of 0·005 *M* ClO_2' and 125 mg per litre of Na oxalate (to complex Fe which catalyses the decomposition of ClO_2') in a total vol. of 40 ml against concn. of aldose. The ClO_2' is determined iodimetrically after aerating the soln. to remove ClO_2. Results obtained by the photometric measurement of the ClO_2 formed in the reaction are compared. Theoretical corrections are suggested and discussed. Determinations of maltose, lactose and melibiose are made. The effect of ClO_2' on non-reducing sugars and sugar acids is shown to be negligible. E. J. H. BIRCH

968. **The separation and identification of normal aliphatic aldehydes and methyl ketones.** P. J. G. Kramer and H. van Duin (*Rec. Trav. Chim. Pays-Bas*, 1954, **73** [1], 63–67).—Mixtures of *n*-aliphatic aldehydes and *n*-alkyl methyl ketones are effectively separated as 2 : 4-dinitrophenylhydrazones by partition chromatography on silica gel by means of nitromethane as the immobile phase and light petroleum as the mobile phase. C_1 to C_{12} homologues are completely separated and C_{12} to C_{18} homologues when differing by two C atoms; they are characterised by their true retention vol. (measured retention vol. minus threshold vol.). Quant. measurements are made via ultra-violet absorption maxima density measurements on conc. eluted material dissolved in $CHCl_3$. D. E. BLENFORD

969. **Separation of volatile acids by paper chromatography.** H. S. Burton (*Nature*, 1954, **173**, 127).—A method is described for locating the position of acid spots on chromatograms of volatile fatty acids prepared by the method of Hiscox and Berridge (*Brit. Abstr. C*, 1951, 252), which overcomes difficulties caused by the background colour often developed. I. JONES

970. **The resolution of mixtures of C_{16} to C_{24} normal-chain fatty acids by reversed-phase partition chromatography.** M. H. Silk and H. H. Hahn (*Biochem. J.*, 1954, **56** [3], 406–410).—Even-numbered straight-chain fatty acids from C_{16} to C_{24} are separated and estimated semi-quant. by chromatography on a column of "Hyflo Super-cel" made non-wetting by treatment with dimethylchlorosilane vapour. Aq. acetone is used as the solvent and purified medicinal paraffin as the stationary phase. A special procedure for packing the columns is necessary to avoid the formation of air bubbles. A siphon, which can be heated electrically if necessary, delivers 1-ml samples of eluate, which are titrated with methanolic KOH in an atm. of acetone-saturated N; bromothymol blue indicator is used. Recoveries are $\simeq$ 75 per cent. for C_{16}, C_{18}, C_{20} or C_{22} acids, and $\simeq$ 60 per cent. for the C_{24} acid. C. E. SEARLE

971. **The determination of hydroxyl, ketone and ester groups in autoxidised fatty esters and related compounds by infra-red spectroscopy.** N. H. E. Ahlers and N. G. McTaggart (*Analyst*, 1954, **79**, 70–76).—Infra-red spectroscopic methods for quant. determination of hydroxyl, ketone and ester groups in autoxidised or co-polymerised fatty esters and related compounds are described. The intensity measurements in solutions sufficiently dilute for the degree of association to be negligible at the wavelengths of the characteristic absorption bands are related to those of suitable reference compounds (methyl ricinoleate, stearate and oxostearate). With ketone-group determinations correction for the general absorption of the ester group is necessary. The methods are simple, rapid and applicable to $\simeq$ 20-mg samples, which can be recovered unchanged afterwards. A. O. JONES

972. **Titration of weak bases in acetic anhydride solvent mixtures.** J. S. Fritz and M. O. Fulda (*Anal. Chem.*, 1953, **25** [12], 1837–1839).—The use of acetic anhydride solvent mixtures in the titration of tertiary amines and alkali metal salts is discussed. For most of the amines and salts investigated, the presence of acetic anhydride increases the sharpness of the break at the end-point as water is removed from the solvent and titrant. The method cannot be used for the determination of primary and secondary amines as excess of acetic anhydride is present during the titration but these amines do not interfere in the titration of other bases, provided they are completely acetylated by heating with acetic anhydride beforehand. The method is also applicable to numerous heterocyclic compounds,

such as those of the purine, pyridine, pyridone and thiazole types. Nitromethane - acetic anhydride mixtures often given sharper end-points than those of acetic acid and acetic anhydride. G. P. Cook

973. **Functional organic micro-analysis based on the determination of physical constants. Identification of amino-acids.** A. Lacourt, G. Sommereyns, C. Francotte and N. Delande (*Mikrochim. Acta*, 1953, [4], 305–331).—The amino-acids can be identified by determining certain physical constants of single crystals on the microscope stage. Particularly, the m.p. of binary eutectic mixtures is specific, and a list of the m.p. of such mixtures is given. Most of the m.p. lie below 150° C so that the amino-acid is not decomposed. The refractive index can also be used for identification. Each determination can be made in 1 min. A. J. Mee

974. **Micro-determination of physical constants of amino-acids.** G. Sommereyns (*Mikrochim. Acta*, 1953, [4], 332–344).—Determination of the m.p. of amino-acids by the Kofler method gives values that often differ widely from those recorded in the literature. Specimens of the same substance from different sources often have different m.p., but the m.p. of eutectic mixtures of these acids are concordant. The ease with which the amino-acids decompose makes it difficult to determine their m.p. accurately. Aspartic and glutamic acids can be identified by the m.p. of their eutectics with β-alanine, hydroquinone, thiourea, salophen, hippuric acid, saccharin or atophan [cinchophen]. The m.p. determined by the microscope is often higher than that determined on a heating block. *nor*Leucine, leucine and *iso*leucine can be detected from the m.p. of their eutectics with urea, di-iodohippuric acid, or 2:4-dinitrodiphenylhydrazine. For *iso*leucine, mannitol and thiourea are also suitable. β-Phenylalanine and tyrosine can be detected by the m.p. of their eutectics with oxalic, malonic, succinic, tartaric, ascorbic, *o*- and *p*-hydroxybenzoic, hippuric and di-iodohippuric acids, urea, thiourea, and di-iodohydroxyquinoline. A. J. Mee

975. **Quantitative determination of isomers of O:O-diethyl ethylthio-ethyl thiophosphate.** K. Gardner and D. F. Heath (*Anal. Chem.*, 1953, **25** [12], 1849–1853).—Studies on pure O:O-diethyl O-ethylthio-ethyl thiophosphate (**I**) showed that the physical and toxicological properties of this compound differed from those of the active constituent of the insecticide Systox, which is stated to be **I**. Concurrent partition chromatography on a mixture of radioactive preparations (^{32}P) of **I** and O:O-diethyl S-ethylthio-ethyl thiophosphate (**II**) with the active ingredient from Systox showed the presence of **I** and **II** in the insecticide. It is also shown that **I** isomerises to **II** on heating. A chromatographic method for the determination of **I** and **II** is also described, the solvent being *iso*octane, 80 per cent. saturated with methanol. The eluate fractions are analysed for P by the colorimetric molybdate method. G. P. Cook

976. **Contribution to the determination of organic sulphur derivatives.** B. Gauthier and J. Maillard (*Ann. Pharm. Franç.*, 1953, **11** [7–8], 509–522).—A number of org. S compounds (method of prep. and m.p. are given) are determined in acetic acid soln. by titration with 0·1 *N* bromate - bromide soln., or by back-titration with $Na_2S_2O_3$ after addition of excess of bromate - bromide soln. The number of equiv. of Br consumed and suitable times of reaction for back - titrations are determined for each compound. The thioacetals are titrated directly at 30° to 40° C; 3:3-di(carboxymethylthio)-7:12-dioxocholanic acid (and its methyl ester), 3:3-di(carboxymethylthio)-12-oxocholanic acid, 1:1-di(carboxymethylthio)-1:2:3:4-tetrahydronaphthalene, 1:1-di(carboxymethylthio)-1-phenylethane, di(carboxymethylthio)naphth-1-ylmethane, and glucose dibenzylmercaptal consume 12 equiv. of Br, and lactose dibenzylmercaptal 16 equiv. Br per mol. Direct titration of alkyl sulphides at ordinary temp. affords only sulphoxides but excess of Br (in back titration) leads to sulphones. Direct titration of 1:2-di(carboxymethylthio)ethane consumes 6, of octylthioacetic acid 2, of diphenylmethylthioacetic acid 6, of 1-carboxymethylthiobenzene-2-carboxylic acid 2 and of 1-hydroxyethylthiobenzene-2-carboxylic acid 2 equiv. of Br per mol. These same compounds and benzylthioacetic and benzylthiomalic acids are also titrated by determination of excess of Br after various times of reaction. Thiolic esters react too slowly for direct titration, but by back-titration benzoylthioacetic acid requires 6 equiv. of Br in 20 sec. and 8 equiv. in 35 min., and naphth-2-oylthioacetic acid 6 equiv. in 3 min. The literature of such oxidations is briefly reviewed. E. J. H. Birch

977. **A modified iodimetric determination of organic peroxides.** B. D. Sully (*Analyst*, 1954, **79**, 86–90).—The method presented is similar to that of Lea (*Brit. Abstr. C*, 1947, 132), but the necessity for de-aeration of the reagents and use of an inert atm. are avoided by mixing all the reactants in boiling acetic acid and chloroform soln. After boiling the peroxide in this medium with KI in a simple refluxing apparatus, the mixture is cooled and diluted and the liberated I is titrated with $Na_2S_2O_3$. A. O. Jones

978. **New method for the determination of resorcinol.** C. Stainier and J. Bosly (*J. Pharm. Belg.*, 1953, **8** [9–10], 443–448).—A colorimetric method is described for the determination of resorcinol by its reaction with HIO_3. To 2 to 5 mg of resorcinol in 5 ml of distilled H_2O is added 5 ml of HIO_3 soln. (15 g in 100 ml of H_2O). After 30 min. the yellow-orange coloration is measured against standard soln. on a colorimeter. Benzophenol reacts with IO_3' similarly, but the reaction is less sensitive, and the determination of resorcinol can be performed in the presence of an equal or smaller concn. of benzophenol. N. M. Waller

979. **Determination of phthalic anhydride in crude phthalic anhydride.** M. Struszynski, Z. Bellen and N. Bellen (*Przem. Chem.*, 1953, **32** [5], 243–245).—The gravimetric method of Kappelmeier (*Farben Ztg.*, 1935, **40**, 1141) and the oxidimetric method of J. J. Yoffe (*Zavod. Lab.*, 1950, **10**, 1252) have been modified and combined to give a quant. determination of phthalic anhydride in the presence of maleic and benzoic acids, alcohol sol. and alcohol - ether-sol. substances and of compounds that are not pptd. by alcoholic KOH. A sample (≃ 2 g) is dissolved in 10 ml of ethanol, and 25 ml of 0·5*N* ethanolic KOH are added. The soln. is heated to 70° to 75° C for 15 min., then cooled to room temp. and 15 ml of ether are added. After 10 min. the mixture is neutralised with oleic acid (phenolphthalein) and filtered through a G3 or G4 sintered-glass filter. The K phthalate

ppt. is washed 5 times with 5 ml of ethanol - ether (1 + 1), dried at 160° C and weighed after cooling. The aq. solution of the ppt. is transferred to a glass bottle with ground-in stopper, 1 ml of conc. H_2SO_4, 20 ml of 0·1 *N* $KMnO_4$ and, after 3 min., 0·5 g of KI are added. The I liberated is titrated with 0·1 *N* $Na_2S_2O_3$. Any alcohol-insol. matter in the original substance is determined in a separate sample. The percentage of phthalic anhydride (x) is calculated from: $x = \frac{61{\cdot}13G \times 1{\cdot}175\ (V_1 - V_2)}{Q}$ per cent., where Q is the wt. of sample, G is the wt. of ppt., V_1 is the vol. of 0·1 *N* $KMnO_4$ used and V_2 is the vol. of 0·1 *N* $Na_2S_2O_3$ used. The method is suitable for routine tests. Results are $\simeq$ 98·7 per cent. of the theoretical. H. BURSTIN

980. **Studies on polarographic analysis. XVII. Electrolytic reduction of *cyclo*hexanedionedioxime and its application to amperometric titrations.** M. Ishibashi, T. Fujinaga and K. Kawamura (*Bull. Chem. Soc. Japan*, 1953, **26** [9], 513–516).—The amperometric titration of Ni ($\simeq$ 1 to 10 mg) with a 0·0211 *M* aq. soln. of *cyclo*hexanedionedioxime (nioxime) is described; it is found to give best results in ammoniacal medium (0·25 *M* NH_4Cl and NH_3) at an applied e.m.f. of 1·8 V; errors for the Ni are $< \pm 2$ per cent. The reduction mechanism of nioxime is briefly discussed.

D. E. BLENFORD

981. **Alkyl substituted 2:2′-diquinolines and alteration of their spectrophotometric constants following [cuprous] copper chelation.** G. F. Smith and D. H. Wilkins (*Anal. Chim. Acta*, 1954, **10** [2], 139–146).—The absorption spectra of the Cu^I complexes of 2:2′-diquinolyl (max. at 540 mμ, ε 5490), 3-methyl- (520, 4120), 3-ethyl- (525, 3030), 3-propyl- (523, 3500), 3-phenyl- (529, 5610), 3-carbethoxy- (519, 3420), 4-methyl- (547, 6490), 4-phenyl- (553, 8440), 4:4′-dimethyl- (551, 7140) and 4:4′-diphenyl-2:2′-diquinolyls (559, 9020) have been studied. It is noted that for all of these except 3-phenyl-2:2′-diquinolyl, 3-substitution decreases the wavelength of max. absorption and ε, whilst 4-substitution increases them. The reasons for the effects are discussed in relation to the coplanarity and electron distribution of the quinoline residues.

E. J. H. BIRCH

982. **A new reagent for the quantitative determination of thiophen.** J. Giral and E. Jaimes (*Ciencia*, 1953, **13** [4–6], 75–76).—The reagent, a soln. of 0·5 per cent. of selenium dioxide in conc. H_2SO_4, is sufficiently sensitive to detect 3·3 μg of thiophen in 1 ml of benzene. One ml of reagent is shaken with 10 ml of the benzene (or other thiophen-containing oil), the benzene is removed on a water-bath, and a permanent blue colour forms after cooling for 15 min. if thiophen is present. Quant. estimation can be made from a calibration curve by use of a Klett - Summerson photo-colorimeter with a No. 66 red filter. The graph between 3·3 μg and 33 μg is linear. M. TADMAN

983. **The composition [and analysis] of commercial ethylbenzylaniline sulphonic acid.** K. S. Heine, jun. and J. H. Jones (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 923–930).—Infra-red and ultra-violet absorbancy curves are given. The latter can be used to determine the composition of a mixture of any two of the isomers of ethylbenzylaniline sulphonic acid. Commercial samples contain mainly the *m*-isomer, with 9 to 18 per cent. of *p*- and 2 per cent. or less of the *o*-isomer. A. A. ELDRIDGE

984. **Method of characterising gasolines by [separation into] hydrocarbon types.** L. Robert and A. Crozier (*Rev. Inst. Franç. Pétrole*, 1953, **8** [11], 545–549).—A simple and fairly rapid method is described for the characterisation of gasolines. Five hundred ml of spirit are fractionated into 12 clear cuts through a column of at least 30 theoretical plates. Each fraction is then analysed for aromatics by sulphonation, for olefines by the bromine number, and for paraffins and naphthenes by the aniline point of the saturated portion. The method is useful for process control. A. JOBLING

985. **Direct extraction-pyknometer method for oil content of refinery effluents.** W. S. Levine, G. S. Mapes and M. J. Roddy (*Anal. Chem.*, 1953, **25** [12], 1840–1844).—A rapid and accurate method, which is especially applicable to waste waters containing appreciable quantities of volatile material, is described. The oil is extracted with water-saturated CCl_4 and after filtration and separation from entrained water, the extract is weighed in a two-armed pyknometer. The oil content of the sample is then calculated from the difference in wt. between the oil - CCl_4 soln. and an equal vol. of the pure CCl_4, the density of the oil being determined by prior measurement and this value being assumed in the calculation for the effluent sample. The recoveries attained with S.A.E.10 oil and separator oil are 97·5 and 99·8 per cent., respectively, and for kerosene and gasoline (boiling at 100° F initially) the recoveries are 94·3 and 83·6 per cent., respectively. Good recoveries of the same order are attained for mixtures of these oils; the average deviation from known values is < 6 per cent.

G. P. COOK

986. **Determination of naphthalene in wash oil and coke oven gas. Infra-red and ultra-violet spectrometry.** N. J. Klein and G. W. Struthers (*Anal. Chem.*, 1953, **25** [12], 1818–1821).—The method for the determination of naphthalene in wash oil depends on the intensity of the specific naphthalene band at 12·77 μ, slight compensation being required for the absorbance of the wash oil at this wavelength. The solvent used for these samples is 2:2:4-trimethylpentane, the background absorbance of the oil in this medium being fairly linear with its concn. The concn. range is 0·50 to 4·00 per cent. of naphthalene and a sample of wt. is chosen such that the naphthalene content of the *iso*octane soln. is between 0·005 and 0·10 per cent. The most serious interference is from xylene, but benzene, toluene and phenol also interfere slightly. The precision of the method is $\pm$ 3·2 per cent. (P = 0·95), calculated from the analysis of 10 separate portions of a standard containing 2·49 per cent. of naphthalene. The method for coke oven gas involves the scrubbing of the gas with *cyclo*hexane, followed by ultra-violet measurement of the naphthalene - *cyclo*hexane soln. at 309, 311·5 and 318·5 mμ. Naphthalene exhibits an absorption maximum at 311·5 and minima at 309 and 318·5 mμ. The concn. range for the method is 0·001 to 0·005 per cent. of naphthalene and the precision is $\pm$ 1·6 per cent. (P = 0·95), calculated from the analysis of 10 separate portions of a standard containing $\simeq$ 10 mg of naphthalene per 100 ml of *cyclo*hexane. G. P. COOK

987. **Synthetic colours in use in the pharmaceutical and food industries. New methods of extraction, separation, identification and assay. I.** W. Lhoest

(*J. Pharm. Belg.*, 1953, **8** [3–4], 119–145).—The extraction of synthetic dyes from various classes of products is described. Water-sol. and unfixed dyes can be extracted by dyeing wool in soln. acidified to 2 per cent. with tartaric acid. Meat dyes are extracted by destruction of the proteins with pepsin; vegetable dyes are similarly separated by enzyme action on the substrate and subsequent ether extraction of the chlorophyll after acidification to 0·2 per cent. with HCl. Dyes present in starchy foods are separated from gluten by the action of pepsin, and oil dyes are extracted into light petroleum and separated by chromatography on an alumina column. N. M. WALLER

988. **Synthetic colours in use in the food and pharmaceutical industries. New method of extraction, separation, identification and determination. II.** W. Lhoest (*J. Pharm. Belg.*, 1953, **8** [5–6], 260–282).—The separation and identification is described of mixtures of dyes extracted from foods and drugs. The method is the preparation of a two-dimensional chromatogram combining both partition and adsorption chromatography. In the partition method for the first dimensions, the solvent used is *n*-butanol - ethanol - distilled water (2:1:1), for the second dimension either 2 per cent. Na_2CO_3 or *N* aq. NH_3 may be used, the later requiring an atmosphere saturated with NH_3. The dyes can be identified by their R_F values in each dimension. Oil dyes can be identified by extraction on an alumina column, and their identity can be confirmed by paper chromatography with glacial acetic acid. N. M. WALLER

989. **Synthetic colours in use in the pharmaceutical and food industries. New methods of extraction, separation, identification and assay. III.** W. Lhoest (*J. Pharm. Belg.*, 1953, **8** [7–8], 371–391).—Methods for the identification and assay of dyes used in the food and drug industry are described. Chromatographic separation (see Abstract 988 above) effects some identification. This may be confirmed by a paper chromatographic method described in which simultaneously prepared chromatograms of the test dye and those that most closely resemble it are compared; identification by classical reactions is detailed for 19 dyes. Quant. assay of the dyes is possible by comparing the colours of the chromatographically separated extracts with these of standard samples. N. M. WALLER

990. **Paper chromatography of coal-tar colours.** D. H. Tilden (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 802–810).—Colour reactions and chromatographic solvent systems for migration and for separation are tabulated for a number of D & C and FD & C dyes. A. A. ELDRIDGE

991. **The identification of azo dyes by spectrophotometric identification of their reduction products. II. Compounds which give neutral or acidic products on reduction.** L. S. Harrow and J. H. Jones (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 914–923).—Procedure for the separation of reduction products of azo dyes is described. Absorption curves of the products in acid and alkaline soln. are compared with those of possible reduction products. Typical graphs are given, together with a list of products of various azo dyes (*cf. Brit. Abstr. C*, 1952, 254). A. A. ELDRIDGE

992. **The separation of certain anthraquinone dyes by paper chromatography.** L. C. Mitchell (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 943–946).—Mixtures of sulphonated (D & C Green No. 5, monosulphonated D & C Green No. 5 and External D & C Violet No. 2) and unsulphonated (D & C Green No. 6 and D & C Violet No. 2) anthraquinone dyes can be separated in one step by paper chromatography. The paper is impregnated with a solution of soya-bean oil in ether; the mobile phase is 2-methoxy-ethanol and water (4 + 1 by vol.). A. A. ELDRIDGE

993. **[Determination of] lower sulphonated dyes in FD & C Blue No. 1.** M. Dolinsky (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 798–802).—The original tentative method (Methods of Analysis, A.O.A.C., Sixth Edition, 1945), since deleted (Freeman, *J. Ass. Off. Agric. Chem.*, 1950, **33**, 381) is considered to be satisfactory if a 10-mg sample is used and F. D & C Green No. 1 is used as a spectrophotometric standard. A. A. ELDRIDGE

994. **The separation and determination of sulphonated naphthalene intermediates in certifiable coal-tar colours.** L. S. Harrow and K. S. Heine, jun. (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 936–943).—Procedure for the separation from dyes of sulphonated naphthalene intermediates by column chromatography and their subsequent spectrophotometric determination is described. Percentage recoveries of intermediates added to various dyes were: naphthionic acid (89 to 110); 2-naphthol-3:6-disulphonic acid (80 to 110); 2-naphthol-6:8-disulphonic acid (91 to 100); 2-naphthol-6-sulphonic acid (90 to 105) and 2-aminonaphthalene-1-sulphonic acid (96 to 98). A. A. ELDRIDGE

995. **[Determination of] subsidiary dyes in D & C colours. 4-Toluene-azo-2-naphthol in D & C Red No. 35.** L. Koch (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 796–798).—The 4-toluneazo-2-naphthol is separated from the primary dye by means of chloroform, reduced with $Na_2S_2O_4$ and the 4-methylaniline is determined by means of $KBrO_3$ (Koch, *Brit. Abstr. C*, 1951, 9). The results were not truly quantitative. A. A. ELDRIDGE

996. **Studies on coal-tar colours. XIII. D & C Red No. 33.** R. N. Sclar (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 930–936).—Absorption curves in the visible and ultra-violet regions for D & C Red No. 33 (Acid Fuchsin D, Colour Index No. 30) and Chromotrope 2R (Colour Index No. 29) in acid, basic and neutral solutions permit identification of each dye when present alone, but not of a small percentage of the second mixed with D & C Red No. 3 unless the components are first separated by paper chromatography. To prepare the solvent, water was saturated with *iso*pentanol and NH_3 soln. was added to give a soln. (1 + 99 by vol.). A small quantity of an unidentified blue dye was present. A. A. ELDRIDGE

997. **The determination of wool wax, soap and insoluble dirt in wool-scour liquors.** G. R. Edwards, W. W. Mansfield and A. G. Pagels (*Aust. J. Appl. Sci.*, 1953, **4** [4], 579–580).—The sample of liquor (10 ml) is cracked with 6 *N* HCl (2 ml) and the ppt., which is coagulated by heating to 90° to 95° C. for 10 min., is then washed thoroughly with cold water to remove excess of HCl and the shorter-chain fatty acids derived from the suint content of the liquor. The residual fatty acids are extracted from the ppt. with 70 per cent. ethanol and estimated by titration with 0·02 *N* alcoholic NaOH, the wool wax being then extracted from the dried residue

with hot CCl_4. The final residue of dirt is dried at 90° to 95° C and weighed.

[This is Appendix I to the paper on "The recovery of wool wax from wool-scour liquors" by L. F. Evans and W. E. Ewers on pp. 552–578 of the same issue.] J. M. JACOBS

998. **Experiments on the hydrolysis of 610, 66 and 6 nylon.** J. Haslam and S. D. Swift (*Analyst*, 1954, **79**, 82–85).—In earlier work (Clasper *et al.*, *Brit. Abstr. C*, 1950, 15), nylon samples were hydrolysed with 20 per cent. v/v HCl for long periods; for 610 nylon preliminary pptn. of the sample in a finely divided state was necessary. It is now shown that hydrolysis with 26 per cent. HCl in sealed tubes at 130° C is effective. The degree of hydrolysis is determined by potentiometric titration of the reaction mixture. The first end-point indicates the amount of free HCl present and titration is then continued to the second end-point, the difference in titre between the two end-points indicating the amount of the component acid of the nylon liberated and hence the degree of hydrolysis. Nylons 6 and 66 were hydrolysed in 10 and 16 hr. respectively. Nylon 610 was hydrolysed less readily but some improvement (up to 97·7 per cent. in 24 hr.) was effected by mechanical shaking of the sealed tubes during the reaction. With this nylon results were also more variable.

A. O. JONES

999. **Colorimetric determination of anionic detergents.** F. J. Loomeijer (*Anal. Chim. Acta*, 1954, **10** [2], 147–150).—Addition of solutions of anionic detergents acetate-buffered to pH 5·1 to a buffered mixture of human albumin (0·4 per cent.; 4 ml) and bromocresol purple (0·02 per cent.; 2 ml) causes a change in the absorption at 600 mμ. Small quantities of detergent cause an unexplained rise in the absorption, but larger additions cause a fall due to the displacement of dye from the protein complex. A separate calibration curve must be made for each anionic detergent. Results are quoted for Na dodecyl sulphate, Teepol, Aerosol O.T. and di*iso*propylnaphthalenesulphonic acid. The accuracy is $\simeq$ 4 per cent. E. J. H. BIRCH

1000. [**Analysis of**] **hair dyes and rinses. Analysis of mixtures of *p*-aminophenol and *p*-phenylenediamine or 2:5-diaminotoluene.** S. H. Newburger and J. H. Jones (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 784–789).—*p*-Phenylenediamine, 2:5-diaminotoluene and *p*-aminophenol are quantitatively extracted by ether from a conc. NaCl soln. buffered with $NaHCO_3$. After diacetylation a mixture of *p*-aminophenol with either (but not both) of the other substances can be analysed spectrophotometrically with reasonable accuracy.

A. A. ELDRIDGE

1001. [**Analysis of**] **mascaras, eyebrow pencils and eye shadows.** P. W. Jewel (*J. Ass. Off. Agric. Chem.*, 1953, **36**, 789–791).—Procedures for the analysis of certain types of cream mascaras are proposed. A. A. ELDRIDGE

1002. [**Analysis of**] **deodorants and anti-perspirants. Determination of boric acid.** J. E. Clements (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 791–793).—Martin and Hayes' ion-exchange method (*Brit. Abstr. C*, 1952, 293) is used. The average recovery of added boric acid was 99·5 per cent.

A. A. ELDRIDGE

4—BIOCHEMISTRY

INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

1003. **A rapid titrimetric method for determining the water content of human blood.** F. E. Davis, K. Kenyon and J. Kirk (*Science*, 1953, **118**, 276).—Results for water in human blood are rapidly obtained by the Karl Fischer method. One drop of blood (weighed) is discharged into a flask containing 20 ml of anhyd. methanol previously brought to an orange-red end-point with Fischer reagent. The same end-point is attained again by titrating with Fischer reagent. Results compare well with oven-drying. N. E.

1004. **A simple technique for the estimation of radioactive components of plasma after the administration of radioactive iodide.** F. Brown and H. Jackson (*Biochem. J.*, 1954, **56** [3], 399–406).—Methanol precipitates proteins from plasma and liberates thyroxine from protein combination, while Ag_3PO_4 under specified conditions precipitates iodide and thyroxine. A rapid method of quant. estimation of plasma components containing ^{131}I by these procedures is described; it is applied to the analysis of plasma for human patients and rats tested with ^{131}I. The nature of the fractions obtained is checked by chromatography and autoradiography. C. E. SEARLE

1005. **Spectrophotometric determination of carboxyhaemoglobin.** E. J. van Kampen and H. Klouwen (*Rec. Trav. Chim. Pays-Bas*, 1954, **73** [2], 119–128).—The optical density of a soln. containing a mixture of haemoglobin and carboxyhaemoglobin is measured at a point of max. difference of density (D_m) and at a point of equal optical density (D_i); if a is the fraction of the total as carboxyhaemoglobin then D_m/D_i (observed for the soln.) $= a\,(1/x - 1/y) + 1/y$ where $x = D_i/D_m$ for carboxyhaemoglobin and y is that ratio for haemoglobin prepared from blood taken from subjects who have not smoked for 12 hr. It is shown that the Beer-Lambert law applies and that dissociation of carboxyhaemoglobin on dilution does not occur and dissociation on exposure to light is doubtful. A calibration curve of percentage carboxyhaemoglobin against D_m/D_i is obtained by introducing 0·1 ml of blood into 20 ml of 0·1 per cent. aq. NH_3, haemolysing and centrifuging the soln.; 25 mg of $Na_2S_2O_4$ is added to 10 ml of the soln. Optical densities for one 5-ml portion and for another after coal gas has been passed for 3 min. are measured at 540 (D_m) and 579 mμ (D_i). The determinations are carried out similarly. The error is $\simeq$ 1 per cent. for less than 50 per cent. carboxyhaemoglobin and 2 to 3 per cent. for more than 50 per cent. Sources of error in this and other published methods are discussed.

E. J. H. BIRCH

1006. **Precision of a direct-reading flame photometer for the determination of sodium and potassium in biological fluids.** E. R. Holiday and J. R. K. Preedy (*Biochem. J.*, 1953, **55** [2], 214–220).—A direct reading flame photometer for the estimation of Na and K in urine and serum which has been in continuous use for 6 years is described in detail. The flame spectrum has been analysed by spectroscopic measurements and the efficiency of the filter systems has been assessed. It is shown that

analysis of Na and K in simple soln. of NaCl and KCl is reproducible with a high degree of precision, which compares favourably with most chemical methods; the operation of diluting and reading of samples containing 2 to 5 μg of Na or K per ml has a mean standard deviation of ± 0·03 μg per ml. The validity of Na and K estimations in urine and serum by direct comparisons with standard soln. of NaCl or KCl has been assessed by additional experiments, comparison with chemical methods and serial dilutions. Urinary Na and K estimations are free from interference at sample dilutions between 1:300 and 1:1000. At lower dilutions, readings of both Na and K are depressed. Serum Na estimations are free from interference, but serum K estimations (dilution 1:100) are subject to a systematic error of + 13 per cent. of the chemical estimations. Estimations accurate to ± 1·5 per cent. can, however, be made by applying an arithmetical correction. At lower dilutions progressive depression of the K reading occurs.

P. CHAPLEN

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1009. **Polarographic micro-determination of chloride ion in biological fluids.** O. Tělupilová-Krestýnová and F. Šantavý (*Mikrochim. Acta*, 1954, [1], 64–71).—All the known micro-methods for the polarographic determination of chlorides in biological fluids have been examined, and it is concluded that the original direct determination of chloride ion is the most accurate. A. J. MEE

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Determination in blood plasma—Mix 7 ml of fresh blood with 3 drops of 4 per cent. aq. heparin, cool to 20° C and centrifuge at 2500 r.p.m. for 5 min. To 3 ml of clear plasma add 15 ml of 10 per cent. aq. trichloroacetic acid with continuous shaking, filter, and to 15 ml of clear filtrate add 2 ml of conc. H_2SO_4. Heat on a steam-bath for 10 min. and cool to room temp. Mix the soln. with about 1 ml of saturated Br water, set aside for 5 min. and add 1 ml of *N* KBr followed, with shaking, by 4 ml of 1·5 *N* $KMnO_4$ dropwise. The $KMnO_4$ colour must persist for 30 min. after which time the mixture is cooled in ice and the Br, excess of $KMnO_4$ and manganese oxides are removed by the cautious dropwise addition of cold 3 per cent H_2O_2 at 30 sec. intervals. In the presence of much Br as long as 30 min. may be required. Transfer the decolorised soln. to a separator and extract it with 15 ml of cold petroleum spirit. Discard the aq. phase and wash the petroleum-spirit extract with 10 ml of water, then with 10 ml phosphate buffer (pH 7·0) shaking well. Shake

the petroleum spirit, extract with 10 ml of aq. 4 per cent. thiourea soln. in 4 per cent. borax soln. for 5 min. Siphon off most of the yellow aq. layer, centrifuge to clarify and measure the absorption at 450 mμ within 25 min. of extraction.

Determination in urine—Dilute the urine (1 + 9) with distilled water and mix 2 ml with 3 ml of conc. H_2SO_4. Heat the mixture on the steam-bath for 10 min., cool and add 2 ml of Br water. After 5 min. add 1 ml of *N* KBr and 8 ml of 1·5 *M* $KMnO_4$. Proceed as for blood.

Preparation of standard curve—Dilute 2, 4, 6, up to 20 ml of a soln. of citric acid in 0·1 *N* H_2SO_4 containing 10 μg of anhydrous citric acid per ml, to 20 ml with distilled water, add 2·4 ml of conc. H_2SO_4 and heat on the steam-bath for 10 min. Proceed as described for protein-free plasma.

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1014. **The quantitative determination of bile pigments in serum using reverse-phase partition chromatography.** B. H. Billing (*Biochem. J.*, 1954, **56** [4], xxx).—Serum from cases of obstructive jaundice is treated with freshly diazotised sulphanilic acid in the presence of ethanol, and the two resulting diazonium components are separated on columns of silicone-treated kieselguhr by means of a butanol - water system buffered at pH 4·0. R_F values for the quantities used are given. The relationship between these components and the three original serum pigments separated by Cole, Lathe and Billing (*Biochem. J.*, 1953, **55**, xiii) is established. J. M. SEARLE

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As the alkaline phosphatase activity of serum is stable below 0° C, the use of such a serum as standard is recommended. Evidence is presented that the frozen serum is stable for 7 months and that recovery of such activity when the serum is added to a fresh sample is good.

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1017. **The presence of potassium as a source of inaccuracy in the chemical estimation of sodium in urine.** E. H. Aitken and J. R. K. Preedy (*Biochem. J.*, 1953, **55** [2], 211–213).—A modification of the zinc uranyl acetate method (Dreguss, *Biochem. Z.*, 1939, **303**, 69) was found to give high results for Na in urine especially in urines of low Cl′ content. This was shown to be due to co-pptn. of K. This interference depends on the ratio of Na to K and is independent of the absolute amounts of K present. N. E.

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1019. **Methods of estimation of adrenal cortical steroids with tetrazolium salts.** C. Chen, J. Wheeler and H. E. Tewell (*J. Lab. Clin. Med.*, 1953, **42** [5], 749–757).—3:3′-Dianisole-bis-4:4′-(3:5-diphenyl) tetrazolium chloride (blue tetrazolium or BT) is reduced in alkaline soln. by the five biologically active cortical steroids, deoxycorticosterone, corticosterone, 17-hydroxydeoxycorticosterone, hydrocortisone and cortisone, to give a bluish-red coloration that obeys Beer's law. 2:5-Diphenyl-3-(*p*-iodophenyl)tetrazolium chloride gave, under similar conditions, a pure red colour that was stable over many hours. The compound was not investigated extensively because it is unavailable commercially. The alkali and solvents used influenced the extent, rate of colour formation and its stability. Benzyltrimethylammonium hydroxide, tetramethylammonium hydroxide, tetra-ethylammonium hydroxide, choline and NaOH may be used as alkalis; the last two were studied. Ethanol is used as solvent for organic bases; the purity is not critical. When NaOH is used the purity of the methanol is critical, and the pH must be adjusted by pyridine - HCl for stability of colour. Details are given for the procedure with NaOH; those for that with choline are as follows. *Procedure*—Mix 1·8 ml of the ethanolic soln. of the steroid with 0·1 ml of choline reagent (dilute 2 ml of 50 per cent. choline to 100 ml with ethanol) and 0·1 ml of BT reagent (0·5 per cent. BT in redistilled ethanol), and set aside in the dark for two hr. Then add 5 ml of redistilled ethanol and determine the optical density at 515 mμ. D. C. M. ADAMSON

1020. **A comparison of the tetrazolium and other methods of steroid estimation applied to urine.** J. Wheeler, S. Freeman and C. Chen (*J. Lab. Clin. Med.*, 1953, **42** [5], 758–765).—Methods selected for comparison were those of Heard *et al.* (*J. Biol. Chem.*, 1946, **165**, 687) based on molybdophosphate reduction, Talbot *et al.* (*J. Biol. Chem.*, 1945, **160**, 535) based on copper reduction, Hollander *et al.* (*Endocrinology*, 1951, **49**, 617) based on formaldehyde formation and the tetrazolium method (see Abstract 1019). The best and most reproducible recovery of steroids from urine was by chloroform extraction at pH 1. Continuous extraction with methylene chloride for 24 hr. gave results some 10 per

cent. higher. In 34 human subjects the average excretion found by each of the above methods, calculated as mg per 24 hr., was 1·70, 1·59, 1·35 and 1·54 for males, and 1·53, 1·43, 1·20 and 1·34 for females, all expressed as deoxycorticosterone. Details are given for the extraction and purification of the steroids from urine. The tetrazolium method was best. D. C. M. ADAMSON

1021. **Chromatographic separation of oestrone, oestradiol and oestriol.** J. Bitman and J. F. Sykes (*Science*, 1953, **117**, 356–358).—A method suitable for routine use for the determination of the three oestrogens in blood in quantities of $\simeq$ 3 to 8 μg per litre is described. The absorbent is Celite - NaOH, which effectively separates the three oestrogens. The oestrone and oestradiol can be eluted separately with benzene but the oestriol is retained on the column. CO_2 is bubbled through the column to neutralise the NaOH; the oestriol is then eluted with benzene. N. E.

1022. **Some errors in the colorimetric estimation of oestriol, oestrone and oestradiol by the Kober reaction.** W. S. Bauld (*Biochem. J.*, 1954, **56** [3], 426–434).—Solvent residues are found to cause 15 to 40 per cent. loss of colour in the Kober determination of oestrogens in human urine, as modified by Brown (*J. Endocrinol.*, 1952, **8**, 196), and sources of error in the method are therefore investigated. Failure (of oestriol only) to form the initial yellow complex is due to sulphonation of the quinol, and can be overcome by adding quinol immediately before colour development. The subsequent conversion of the yellow complex to pink is modified to prevent excessive oxidation which causes fading. Variations in the quality of the reagent-grade H_2SO_4 used are eliminated by addition of traces of $NaNO_3$ and quinone. With these changes, solvent residues have no effect on the colour production and Beer's law is obeyed over a suitable range. C. E. SEARLE

1023. **Chemical assay of gonadotrophin in urine.** A. C. Crooke, W. R. Butt, J. D. Ingram and L. E. Romanchuck (*Lancet*, 1954, **i** [8], 379–383).—Gonadotrophins A and B are extracted from 100 ml of urine by adsorption on kaolin, seperated and purified by elution with 0·002 *M* disodium hydrogen phosphate and 0·02 *M* tribasic sodium phosphate, respectively. Three tests are then applied: (*i*) the ninhydrin reaction for free amino groups, (*ii*) the orcinol reaction for hexose and (*iii*) the polarographic method for proteins (Millar, *Biochem. J.*, 1953, **53**, 385). The results are compared with standards prepared from purified gonadotrophins. N. E.

1024. **A colorimetric method for the determination of the principal metabolites of nicotinic acid in human urine.** W. I. M. Holman (*Biochem. J.*, 1954, **56** [3], 513–520).—A colorimetric method for the determination in urine of the main metabolites, the acid amides, of nicotinic acid is developed. It is based on the conversion of the carboxyamide into an amino group by heating with hypobromite, followed by diazotisation and coupling of the amino compound formed to give an azo dye. When the excess of hypobromite is suitably controlled, and N-(1-naphthyl)ethylenediamine dihydrochloride is used as the coupling agent, azo colours can be produced from N-methyl-2-pyridone-5-carboxyamide, N-methyl-2-pyridone-3-carboxyamide, N-methylnicotinamide and nicotinamide. Optimum conditions for colour production are determined and a method is developed for the determination of 1 to 50-μg amounts of each of the colour-producing substances. The removal from urine of substances that interfere with the colour reaction is investigated, and procedures are described which are suitable for the determination of N-methyl-2-pyridone-5-carboxyamide and N-methylnicotinamide in human urine. The urinary excretions of nicotinic acid metabolites by three adult subjects were determined before and after administration of quinolinic acid. The results show evidence of no more than a slight conversion of quinoline into nicotinic acid. I. JONES

1025. **Fluorimetric determination of cholic acid.** M. Pesez (*Ann. Pharm. Franç.*, 1953, **11** [11], 670–674).—The sample containing 20 to 80 μg of cholic acid is diluted to 1 ml with water and 5 ml of H_3PO_4 (sp. gr. 1·61) is added. After heating for 5 min. on a water-bath and cooling in ice - water, 5 ml of *n*-butanol are added, and the soln. is kept in iced water for 10 min. The fluorescence in u.v. (Wood's) light is then measured immediately. The intensity of the fluorescence is extremely sensitive to temperature, but the action is specific, so estimations may be made in biological liquids. The mechanism of the method is discussed. E. J. H. BIRCH

1026. **Chromatographic separation and analysis of mixtures of pyruvic, oxalacetic and alpha-ketoglutaric acids.** F. A. Isherwood and D. H. Cruickshank (*Nature*, 1954, **173**, 121–122).—The method of Cavallini *et al.* (*Nature*, 1949, **163**, 586; 1949, **164**, 792) for the analysis of α-keto-acids in biological extracts, which is based on the chromatographic separation of the 2:4-dinitrophenylhydrazones and the colorimetric estimation of the separated hydrazones, is modified to eliminate the uncertainty arising as a result of the discovery that stereoisomeric hydrazones are formed. The properties of the isomeric hydrazones are discussed. I. JONES

1027. **The chromatographic separation of progesterone and testosterone.** J. Carol (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 1001–1004).—Testosterone is more sol. in dil. ethanol than progesterone. Sharp separation can be effected by chromatographic partition, by use of 80 per cent. ethanol as the immobile solvent on Celite, and *iso*-octane as the mobile solvent. A. A. ELDRIDGE

1028. **Reactions of aromatic nitro compounds with active methyl, methylene and methine groups in presence of base. Determination of creatinine with alkaline 3:5-dinitrobenzoate.** J. J. Carr (*Anal. Chem.*, 1953, **25** [12], 1859–1863).—The conditions of optimum sensitivity and colour stability in the sodium 3:5-dinitrobenzoate method for determining creatinine are given, and an improved method is recommended. Ten ml of a neutral soln. of the sample are mixed with 2 ml of alkaline dinitrobenzoate reagent [0·15 *M* sodium 3:5-dinitrobenzoate in 0·15 *M* NaOH) and the soln. is set aside for 1 hr. The absorption is then measured at 500 mμ and the creatinine content is calculated from the absorption of a standard treated in the same manner. The method is suitable for the determination of 0·1 to 1·0 mg of creatinine in 10 ml of sample. The error is $< \pm$ 1 per cent. G. P. COOK

1029. **Determination of mono-amino mono-carboxylic acids by quantitative paper chromatography.** A. R. Kemble and H. T. Macpherson

(*Biochem. J.*, 1954, **56** [4], 548–555).—The amino-acids (each containing 0·08 to 0·25 mg of N) are separated from acidic and basic amino-acids by electrophoresis, and separated chromatographically in a wide band on filter-paper by means of 80 per cent. propanol as solvent. They are located on spraying with an ethanolic soln. of bromothymol blue, formaldehyde and alkali, cut out and eluted into Warburg flasks. They are then determined by measurement of the CO_2 formed on oxidation with chloramine-T in a Warburg respirometer. The method is applied to synthethic mixtures of amino-acids and to protein hydrolysates. Special procedures are necessary for mixtures of amino-acids not separated on the chromatogram.

C. E. SEARLE

1030. **Phospholipids. II. Estimation of amino nitrogen in intact phospholipids.** C. H. Lea and D. N. Rhodes (*Biochem. J.*, 1954, **56** [4], 613–618).—The ninhydrin method of Moore and Stein (*J. Biol. Chem.*, 1948, **176**, 367), modified to maintain the lipids in solution, is used as a rapid and sensitive method for the estimation of phosphatidylethanolamine (1 to 5 of μg N) in the presence of much larger quantities of non-amino phospholipids [*e.g.*, phosphatidylcholine (up to 1000 μg of N)], without necessitating preliminary hydrolysis. Phosphatidylcholine as well as phosphatidylethanolamine liberates gas in the van Slyke manometric procedure for the estimation of amino N owing to reaction of its component unsaturated fatty acids with nitrous acid. Hence this method can be applied only empirically to unhydrolysed phospholipids with a reaction time chosen for the particular material to agree with a determination on the free hydrolysate freed from fatty acids. It is much less sensitive than the ninhydrin method. I. JONES

1031. **Aromatic aldehydes as specific chromatographic colour reagents for amino-acids.** G. Curzon and J. Giltrow (*Nature*, 1954, **173**, 314–315).—Useful colour reactions of aromatic aldehydes with amino-acid spots on paper chromatograms are described. Phthalaldehyde in acetone (0·2 per cent.) to which urea (0·2 per cent.) has been added gives a purple colour with glycine. Phthalaldehyde also give a specific colour with taurine. Terephthalaldehyde (0·2 per cent. in acetone) is a specific and sensitive reagent for histidine, tryptophan and tryptamine. The addition of 10 per cent. acetic acid enhances the colours with tryptophan and tryptamine but not with histidine. N. E.

1032. **The detection of serine and threonine by the 1:2-dinitrobenzene-enediol reaction.** W. R. Fearon and W. A. Boggust (*Analyst*, 1954, **79**, 101–102).—Compounds containing adjacent -OH and $-NH_2$ groups (*e.g.*, serine and threonine) are de-aminated to enediols by alkaline hypochlorite and these enediols yield a violet colour with 1:2-dinitrobenzene. To 1 ml of the neutral soln. containing $\geq$ 1 mg of the amino-acid are added 2 ml of 0·02 per cent. aq. dinitrobenzene soln., 5 drops of 20 per cent NaOH soln. and 3 to 6 drops of 1 per cent NaOCl soln. At room temp. a violet colour develops in $\simeq$ 10 min. A control containing no NaOCl should be tested simultaneously to detect interference from preformed enediols. Serine is more reactive with hypochlorite than threonine and gives the reaction with 0·1 *N* NaOH in 30 min., threonine not reacting under these conditions. Interference by reducing sugars can be avoided by preliminary titration with Fehling's soln. Interference from ascorbic acid and other highly reactive enediols can be avoided by preliminary titration with iodine in dil. acid soln. An explanation of the mechanism of the reaction is suggested.

A. O. JONES

1033. **Determination of total protein in small quantities of spinal fluids: confirmation of the accuracy of a colorimetric method using Folin-Ciocalteu reagent in the presence of copper.** R. K. Waldman, L. A. Krause and E. K. Borman (*J. Lab. Clin. Med.*, 1953, **42** [3], 489–492).—The colorimetric method for total protein in spinal fluid (Daughaday *et al. Brit. Abstr. C*, 1952, 496) is relatively unaffected by the albumin to globulin ratio of the protein present in contradistinction to turbidimetric methods. The method permits a high degree of accuracy ($\pm$ 10 to 15 per cent.) on samples as small as 0·2 ml. D. C. M. ADAMSON

1034. **Continuous direct photometry of dyed materials in filter-paper, with special reference to the estimation of proteins separated by electrophoresis.** E. M. Crook, H. Harris, F. Hassan and F. L. Warren (*Biochem. J.*, 1954, **56** [3], 434–444).—An apparatus and technique for estimating dyed proteins separated by electrophoresis on filter-paper are described. The paper, immersed in fluid, is carried on a trolley driven by a synchronous motor. The base of the apparatus carries the light source, filters and a selenium photo-cell connected to a polarograph amplifier and recording milliammeter. Large deviations from Beer's law are found, but these can be estimated and compensation can be made. Results by use of the paper and "classical" methods of electrophoresis are discussed.

C. E. SEARLE

1035. **Photometric estimation of deoxyribonucleic acid in animal tissues.** C. Fersini (*Boll. Soc. Ital. Biol. Sper.*, 1953, **29** [8], 1624–1625).—Fresh animal tissue is finely ground and 250 mg is weighed into a centrifuge tube. After being washed with 5 ml of water the sample is centrifuged at high speed for 5 min.; this washing procedure is repeated. Globulin is thus removed. Hydrolysis is carried out with 5 ml of *N* HCl at 60° C for 10 min. After centrifuging, the supernatant liq. is removed, treated with 10 ml of 0·1 per cent. methyl green and left in the thermostat at 37° C for 90 min. The liquid is filtered cold, and the colour measured on a Leitz photometer with a No. 520 filter.

F. R. HARRIS

1036. **A note on the estimation of sphingomyelin in nervous tissue.** R. M. C. Dawson (*Biochem. J.*, 1954, **56** [4], 621–624).—In the estimation of sphingomyelin in brain tissue by the method of Schmidt *et al.* (*J. Biol. Chem.*, 1946, **166**, 505) lower values are obtained when the lipids in the tissue are initially precipitated with trichloroacetic acid (**I**) before they are extracted with solvents. These are shown to be caused by a loss during the precipitation of non-sphingomyelin lipid P, which becomes insoluble in **I** after alkaline hydrolysis and consequently is estimated as sphingomyelin. Even after the precipitation of brain lipids with **I**, the molar ratio of choline to phosphorus of the alkali-stable sphingomyelin is less than unity, indicating that the above method is unsuitable for the accurate estimation of this phospholipid. I. JONES

1037. Comparison of methods for determining proteolytic activity. C. Bowlby, H. Tucker, Byron S. Miller and John A. Johnson (*Cereal Chem.*, 1953, **30** [6], 480–491).—The proteolytic activity of various enzyme prep. determined by (*i*) the Ayre - Anderson digestion of haemoglobin with either Kjeldahl or spectrophotometric determinations of sol. N (Abbott, Miller and Johnson, *Arch. Biochem.*, 1952, **38**, 85), (*ii*) a modified bromosulphalein method (Grief, *Proc. Soc. Exp. Biol. Med.*, 1950, **75**, 813) and (*iii*) a formal titration method (Koch, "Practical Methods in Biochemistry," Third Edition, The Williams and Wilkins Co., Baltimore, Md., 1941) was similar to that given with the Farinograph method (Miller and Johnson, *Cereal Chem.*, 1953, **30**, 471). Methods involving gluten digestion and milk clotting gave different results. S. C. JOLLY

See also Abstracts 899, 1045.

Drugs

1038. Determination of alkaloids in organic solution. H. Wachsmuth (*J. Pharm. Belg.*, 1953, **8** [1–2], 76–80).—Qual. and quant. reactions of alkaloids in organic solvents with metallic salts and other compounds are discussed, showing a wide range of potential analytical methods. Addition compounds with various alkaloids and their salts are formed by $HgCl_2$, $SbCl_3$, HgI_2 in KI, BiI_3, SbI_3, $MnCl_2$ in KI, $UO_2(NO_3)_2$ and the $[Co(CNS)_4]''$ group. Reinecke's salt, picric acid and flavianic acid also ppt. many alkaloids. Flavianic acid forms crystalline ppt. with ephedrine 1:25,000, codeine 1:500,000, papaverine 1:500,000, strychnine 1:5,000,000, ethylmorphine, hyoscyamine, quinine and the hydrochlorides of pilocarpine and pervitine. Results have been satisfactory with this reagent. N. M. WALLER

1039. Determination of alkaloids in organic solution. Action of flavianic acid. H. Wachsmuth (*J. Pharm. Belg.*, 1953, **8** [5–6], 283–289).—Flavianic acid forms crystalline ppt. with many alkaloids, pseudo-bases and amino-acids at low concn., its sensitivity for most alkaloids being between 1:1,000,000 and 1:5,000,000. The nature of these ppt. for over 40 alkaloids is given. The method is used for the quant. determination of alkaloids and pseudo-bases. The alkaloid is dissolved in 10 to 15 ml of ether and to the soln. is added 70 ml of an ethereal soln. containing twice the required quantity of flavianic acid first dissolved in 1 to 3 ml of ethanol. The vol. is adjusted to between 120 and 150 ml with ether, when pptn. occurs. N. M. WALLER

1040. Paper partition microchromatography in phytochemical analysis. Application to the study of some Congo *Strychnos* species. I. Study of non-alkaloidal constituents. F. Jaminet (*J. Pharm. Belg.*, 1953, **8** [7–8], 339–370).—Aqueous alcoholic solutions of *Strychnos* samples are separated into groups by pptn. with Pb acetate and basic Pb acetate. The filtrate contains the sugars and the ppt. contains the organic acids and flavonic pigments, which are further separated by subsequent extractions with ether and ethyl acetate. The members of these groups are identified by paper chromatography. A soln. of the sugars in ammoniacal phenol, or acetic acid and ethyl acetate is used to prepare a chromatogram on Whatman filter-paper, the developing agent being a mixture of phthalic acid and aniline in aqueous *n*-butanol. The flavanol soln. in aq. 5 per cent. $NaKCO_3$ is applied to paper, which is studied under u.v. light to characterise the pigments present. Caffeic and igosauric acids can be similarly identified under u.v. light or by development of the chromatogram by means of an aq. soln. of $FeCl_3$ and $K_3Fe(CN)_6$. For other organic acids such as malic or citric acid, the developing agent is a soln. containing bromophenol blue in 0·1 *N* alcoholic NaOH. A qualitative study of the sugars, acids and flavanols present in the *Strychnos* studied is included. N. M. WALLER

1041. Paper partition microchromatography in phytochemical analysis. Application to the study of some Congo *Strychnos* species. II. Study of the alkaloidal constituents. F. Jaminet (*J. Pharm. Belg.*, 1953, **8** [9–10], 449–473).—The separation and characterisation by paper partition chromatography of the alkaloid constituents of several *Strychnos* species are described. Solvent phases used include *iso*butanol, pentanol, *iso*propanol, conc. HCl, 5 per cent. acetic acid, 17 per cent. NH_3 soln. and distilled water. The developing agents are Dragendorff's reagent (prepared by mixing 850 mg of $Bi(NO_3)_3$ in 10 ml of glacial acetic acid and 40 ml of water with 8 g of KI in 20 ml of water) and an iodoplatinic reagent (prepared by mixing 1 ml of 10 per cent. $PtCl_4$ soln. with 25 ml of 4 per cent. KI soln., and diluting this mixture to 50 ml). These reagents permit the characterisation of spots containing 10 to 20 μg of alkaloid. By this method with separation on alumina columns, three alkaloids have been separated in the leaves of *Strychnos Icaja* and their colour reactions, absorption spectra and empirical formulae have been studied. A method for the determination of 20 to 80-μg quantities of strychnine after chromatographic separation is described; it has been successfully applied to the determination of strychnine in powder and tincture of nux vomica. N. M. WALLER

1042. [Determination of] quinine. Separation of quinine and strychnine. D. J. Miller (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 708–713).—The average recovery of quinine from a mixture with strychnine by the use of the modified A.O.A.C. (gravimetric) method (Miller, *J. Ass. Off. Agric. Chem.*, 1950, **33**, 192) in collaborative work was 100·1 per cent.; that of strychnine was 98·8 per cent. Brucine is extracted with the quinine. In a spectrophotometric method, which requires only 4 mg of strychnine and 10 mg of quinine, quinine is determined by its absorption at 347·5 mμ and strychnine after reduction with Zn - Hg and treatment with $NaNO_2$, by measuring absorption at 530 mμ. Recoveries were 99·1 to 100·3 and 90·1 to 95·2 per cent., respectively. A. A. ELDRIDGE

1043. Chemical determination of the alkaloid content of ergot. M. Pöhm (*Mitt. Chem. ForschInst. Ost.*, 1953, **7** [6], 121–123).—The principles underlying a colorimetric method (at present being worked out) for the quantitative determination of the water-soluble and water-insoluble alkaloids of ergot are described. W. GOOD

1044. Fluorimetric determination of gitoxin. J. F. A. Fruytier and J. A. C. van Pinxteren (*Pharm. Weekbl.*, 1954, **89** [7–8], 99–103).—A mixture of glycerol and HCl (*cf.* Jensen, *Brit. Abstr. C*, 1953, 24) is an unsatisfactory solvent, but satisfactory fluorimetric results are obtained after dissolving the dry prep. (representing an aliquot portion of an aq. ethanolic extract) in 1 ml of 96 per cent. v/v ethanol, mixing the soln. with

10 ml of (1 + 1) glycerol and 38 per cent. HCl, and setting aside for 1 hr. Digitoxin does not interfere with the determination. The intensity of the fluorescence is proportional to the gitoxin content. Commercial samples of digitoxin contained 5·8 to 15·6 per cent. of gitoxin. In a sample of digitalis leaf, ≃ 67 per cent. of the glycosides were of the B (gitoxin) group. P. S. ARUP

1045. **The quantitative determination of adrenaline and *nor*adrenaline in mixtures.** H. Persky and S. Roston (*Science*, 1953, **118**, 381–382).—Differences in spectral characteristics of the fluorescence emission of the condensation products with ethylenediamine form a basis for the quant. differentiation of adrenaline and *nor*adrenaline in mixtures. N.E.

1046. **Studies on antibacterial activity and bioassay method of Ilotycin (erythromycin).** Y. Hara, S. Watanabe and A. Tamaki (*Acta Med. Biol., Japan*, 1953, **1** [1], 7–12).—The antibiotic Ilotycin (erythromycin), like penicillin, is active against gram-positive and gram-negative cocci, against gram-positive bacilli and some gram-negative bacilli (*e.g., H. pertussis*, Brucella-groups), and also against rickettsiae and large viruses. The antibacterial activity at pH 7·0 is twice as strong as that at pH 6·0, and the activity at pH 8·0 twice as strong as that at pH 7·0. The antibiotic resembles streptomycin in that the activity is stronger in alkaline media. Examination of the bioassay of the antibiotic shows that the diffusion method of Miyamura *et al.* (*J. Antibiotics*, 1950, **3**, 411) is the best, and that the optimal pH of the assay broth is 7·5 to 8·0. I. JONES

1047. **A method for the determination of penicillinase.** R. G. Tucker (*Nature*, 1954, **173**, 85).—A method for the determination of penicillinase is described. The method is based on the formation of penicilloic acid from penicillin in the presence of penicillinase. The reaction is followed by the estimation of penicilloic acid iodimetrically. The iodine uptake by penicilloic acid is not stoicheiometric, 6 to 9 equivalents of iodine being absorbed per mole of penicilloic acid according to the conditions. A standard procedure is described whereby reproducible results can be obtained. I. JONES

1048. **Assay of papain preparations.** P. H. Mars (*Pharm. Weekbl.*, 1954, **89** [7–8], 97–98).—The method of the French Codex is examined, and the following improved method is proposed: 1 ml of the filtered extract obtained by soaking 75 mg of the prep. for 1 hr. in 5 ml of water at room temp. is added to a suspension of 1·25 g of specially purified casein in 25 ml of water, and the mixture is incubated for 2 hr. at 70° C, cooled and filtered. A mixture of 10 ml of the filtrate and 25 ml of a neutral (to phenolphthalein) (1 + 2) mixture of formaldehyde soln. and water is titrated with 0·02 *N* NaOH, of which ≮ 20 ml should be required for neutralisation. P. S. ARUP

1049. **Contribution to the quantitative analysis of mixtures of antineuralgics and antipyretics.** C. Stainier (*J. Pharm. Belg.*, 1953, **8** [1–2], 3–11).—The separation into groups of previously identified mixtures of antineuralgics and antipyretics can be effected by extraction at various pH values. Final separation of binary mixtures is avoided by the use of direct quant. methods for the analysis of these mixtures. Phenazone with caffeine, acetanilide, Exalgin or phenacetin can be determined by the iodimetric method of Bougault - Kolthoff or by titration with picric acid. In mixtures with acetanilide, phenacetin or Exalgin may be pptd. as their tetra-iodo complexes and the acetanilide extracted with ether from the filtrate. Exalgin can be pptd. satisfactorily with $K_4Fe(CN)_6$ in the presence of caffeine, but phenacetin becomes entrained with Exalgin under the required conditions of precipitation, giving unsatisfactory results. N. M. WALLER

1050. **Detection of analgesics and alkaloids by means of sodium tetraphenylboron ("kalignost") and nitro compounds.** R. Fischer and M. S. Karawia (*Mikrochim. Acta*, 1953, [4], 366–374).—In order to identify 13 analgesics and 17 alkaloids, the m.p. of the ppt. of the compounds with sodium tetraphenylboron and the m.p. of the eutectics of these ppt. with acetamide and phenacetin and occasionally also with salophen have been determined. The m.p. of the compounds of the analgesics with picrolinic acid, styphnic acid and trinitrophloroglucinol, and of several eutectics have also been determined. The fission of the compounds with picrolinic acid styphnic acid and trinitrophloroglucinol has been followed quantitatively on an Al_2O_3 column, the solvents used being chloroform or mixtures of chloroform with ethanol or *iso*-propanol. A. J. MEE

1051. **Identification of barbiturates by X-ray analysis. VIII. Aethallymal and the system Aethallymal - Diallymal.** R. Heiz and B. Jerslev (*Dansk. Tidsskr. Farm.*, 1954, **28** [1], 11–18).—The X-ray powder photograph of the stable modification of Aethallymal (5-allyl-5-ethylbarbituric acid) (**I**) is very similar to that of Diallymal (5 : 5-diallylbarbituric acid) (**II**). Identification of solid soln. of **I** and **II** can be performed easily, but mechanical mixtures of the two compounds are more difficult to identify. N. E.

1052. **[Analysis of] Tuinal ®. [Determination of] Amobarbital sodium and Secobarbital sodium.** G. E. Keppel (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 725–730).—Total barbiturate is determined by measuring the absorption at 244 mμ. Secobarbital (quinalbarbitone) sodium is determined bromometrically and Amobarbital (amylobarbitone) sodium by difference. In collaborative work recoveries of 98·7 to 108·0 and 95·7 to 109·3 per cent., respectively, were reported. A. A. ELDRIDGE

1053. **Analytical characteristics of some synthetic curarising agents.** —, Simon-Dorlet (*J. Pharm. Belg.*, 1953, **8** [3–4], 146–155).—A method of characterising synthetic curarising agents is based on the cryst. form of the derived picrates, picrolonates, flavianates and their complexes formed with methyl orange. Further identification is furnished by the refractive indices of the picrate crystals. A method for the quant. determination of certain curarising agents is as follows: to 50 mg of the product in 20 ml of H_2O is added 2·5 g of NaCl and 25 ml of 25 per cent. H_2SO_4. This soln. is mixed with 25 ml of 0·1 *N* I soln. and the vol. is adjusted to 50 ml. After 10 min. the ppt. is filtered off and 25 ml of the filtrate are titrated with 0·1 *N* $Na_2S_2O_3$. The curarising agents used were decamethonium iodide (Syncurine) gallamine (Flaxedil), pentamethonium bromide, azamethonium bromide (Pendiomide)

hexamethonium bromide (Bistrium), tetra-ethyl-ammonium bromide (Tetranium), tetra-ethyl-ammonium chloride (Etamon) and cetrimide (Cetavlon). N. M. WALLER

1054. **Analytical study of the nicotinic acid group [of drugs].** J. Deltombe (*J. Pharm. Belg.*, 1953, **8** [1–2], 59–75).—The results of a qual. study of the reactions of nicotinic acid, nicotinamide, nikethamide and isoniazid are tabulated. The König reaction applied to nicotinic acid and its deriv. is discussed and the prep. of the 10 per cent. soln. of BrCN is as follows. To 5 g of KBr in 25 ml of distilled water in an Erlenmeyer flask is added 7·5 g of Br. The soln. is thoroughly cooled and decolorised with 10 per cent. KCN soln., the vol. of the soln. being adjusted to 100 ml. With this soln. and benzidine various colour reactions are obtained at low dilutions with nicotinic and *iso*nicotinic acids and their hydrazides. The preparation of *iso*nicotinic acid from its hydrazide is discussed and methods are reviewed for the volumetric, spectrophotometric, colorimetric and microbiological assay of the hydrazide. N. M. WALLER

1055. **The determination of *iso*nicotinic acid hydrazide in tablets by ultra-violet spectrophotometry.** J. Carol (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 722–725).—The u.v. spectrophotometric procedure gives accurate results in acid or alkaline soln. A. A. ELDRIDGE

1056. **Analytical study of the antihistamines.** A. Bürgin (*J. Pharm. Belg.*, 1953, **8** [1–2], 12–22).—A modification of the Stas - Otto method is used to separate the antihistamines from prep. containing acetylsalicylic acid, phenacetin, caffeine and other components. Systematic extraction with organic solvents and adjustment of pH allows the antihistamines to be extracted with the alkaloids into ether at pH 8 to 11. The antihistamines are separated chromatographically and identified by the m.p. and n_D of their picrates, and by colour reactions with Reinecke's salt. A table of results for 13 antihistamines is given. N. M. WALLER

1057. **[Determination of] dextro- and racemic-amphetamines.** L. H. Welsh (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 714–722).—Procedure involving acetylation followed by extraction and weighing the acetylamphetamine and determining its specific rotation is detailed. Collaborative results obtained for tablets containing 2 per cent. of a (1 + 1) mixture of D- and DL-amphetamine sulphates with lactose, potato starch, Ca_3PO_4, talc and stearic acid gave an average recovery of 96·8 per cent.; with the D-isomer the recovery was 97·9 per cent. of the theoretical. Alternatively the base can be extracted with ether and determined volumetrically. A. A. ELDRIDGE

1058. **The colorimetric determination of caffeine in tablet mixtures.** R. A. Daoust (*J. Amer. Pharm. Ass., Sci. Ed.*, 1953, **42** [12], 744–746).—A quant. colorimetric method for the determination of caffeine in the presence of aspirin and phenacetin is described. Caffeine is precipitated from dil. HCl soln. with molybdophosphoric acid. The caffeine molybdophosphate is filtered and dissolved in acetone, its absorption being measured at 440 mμ. The method is sensitive and reliable and can be used for samples containing from 1 to 5 mg of caffeine. N. M. WALLER

1059. **[Determination of] rutin in tablets.** Arthur Turner, jun. (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 699–707).—Rutin is extracted with acidified ethanol and absorbances at 338·5, 362·5 and 366·5 mμ are determined. Collaborative results are tabulated and subjected to statistical analysis. A. A. ELDRIDGE

1060. **Soluble aspirin tablets N.F.** G. Raine (*Pharm. J.*, 1954, **172**, 31).—Tests for soluble aspirin and free salicylic acid are prescribed and a method of assay for acetylsalicylic acid content depending on hydrolysis and bromine absorption is suggested. N. E.

1061. **Conspers. Zinc Oxid. et Acid Salicyl. B.P.C.** J. V. Mitchell (*Pharm. J.*, 1954, **172**, 102).—The B.P.C. method for determining salicylic acid gives low results. Acidification before extraction with ether was satisfactory. N. E.

1062. **Application of complexones to pharmaceutical analysis. Determination of calcium salts in medicaments.** E. Leroi and J. -A. Gautier (*Ann. Pharm. Franç.*, 1953, **11** [5], 329–336).—Ca salts of a number of organic acids used pharmaceutically are determined by titration with complexone III (disodium dihydrogen ethylenediaminetetra-acetate) without previous mineralisation, (*i*) directly with murexide as indicator at pH > 12 (NaOH), (*ii*) directly with Eriochrome black T in NH_3 - NH_4Cl buffer or (*iii*) by back-titration of excess of complexone III with standard $MgCl_2$ also in NH_3 - NH_4Cl buffer. In method (*i*), Cu^{++} and large quantities of Mg^{++} interfere, but Ba^{++} and Sr^{++} do not; Ca oxalate is too insol. in NaOH soln. In method (*ii*) Ca oxalate and tartrate are both too insol., and all di- and tervalent cations interfere. Method (*iii*) although requiring three standardised solutions and interfered with by di- and tervalent cations, is more generally applicable, as even Ca oxalate is not re-precipitated on buffering in complexone soln. Ca citrate gives erroneous results by all these methods. E. J. H. BIRCH

1063. **Analytical studies on harvested tobacco leaves.** W. G. Frankenburg, A. M. Gottscho, S. Kissinger, D. Bender and M. Ehrlich (*Anal. Chem.*, 1953, **25** [12], 1784–1797).—A comprehensive analytical procedure for determining the various nitrogenous components in tobacco leaves is described. The procedure starts with a water extract of a tobacco sample and continues with the successive separation and determination of all the nitrogenous components by various analytical means. The separations are achieved by various concentration, precipitation and distillation procedures and include a pptn. with tungstosilicic acid to separate the less volatile alkaloids, certain alkaloid transformation products and other nitrogenous components, which are pptd. with this reagent. The analytical procedures used for the determination of N in these components are discussed and include the Kjeldahl method, a modified Nessler procedure for the determination of NH_3 and a u.v. absorption method for nicotinic acid. The water-soluble components determined are nicotine, free NH_3, amide of glutamine, amide of asparagine, NH_3 from unknown sources, NO_3', non-nicotine alkaloids, oxynicotine, α-amino-acids and nicotinic acid. Determination and separation of unknown nitrogen compounds are also achieved and these are classified according to the separation stages in which they occur. Satisfactory agreement

is obtained between the total-N content, as determined by direct Kjeldahl digestion, and the total-N content obtained by summation of the individual N components, known and unknown, the average deviation being ± 1·23 per cent. calculated from the results of 14 samples.
G. P. COOK

1064. **Collaborative study of methods for analysis of tobacco. Nicotine and moisture.** C. O. Willits, M. Gaspar and J. Naghski (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 1004–1018).—Collaborative results are considered statistically, and variables are evaluated. For determining nicotine the A.O.A.C. tungstosilicic acid gravimetric method and the spectrophotometric procedures give equally precise and accurate values. Conditions affecting or not affecting the results are listed. The use of MgO gives lower values for total alkaloids as nicotine than that of NaOH. A. A. ELDRIDGE

See also Abstracts 964, 1015, 1109, 111, 112.

Foods

1065. **Colour measurement. Application to food quality grades.** D. B. Hand, W. B. Robinson, T. Wishnetsky and J. R. Ransford (*J. Agric. Food Chem.*, 1953, **1** [20], 1209–1212).—Theoretical considerations of the physical measurements of reflectance colours and the methods of subjective grading are discussed. E. G. BRICKELL

1066. **The determination of tin in canned foods.** D. Dickinson and R. Holt (*Analyst*, 1954, **79**, 104–106).—The previously charred sample (5 to 10 g) is ignited to ash at 600° C, fused with Na_2CO_3 and KCN (3 + 1), boiled with HCl and filtered. Copper is extracted from the filtrate with diethylammonium diethyldithiocarbamate (prep. described) in $CHCl_3$. An aliquot of the Cu-free soln. is treated with dil. HCl, a few drops of a Teepol soln. and 0·3 ml of dithiol reagent (prep. described) and heated in a bath of boiling water for 2 min. The concn. of the red soln. is then measured absorptiometrically. The reagent used for extraction of Cu is better than Na diethyldithiocarbamate as it does not extract Sn even when present in amount greater than that equiv. to the Cu present. A. O. JONES

1067. **Benzene hexachloride in foods. Determination of total BHC and β-isomer.** A. K. Klein (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 589–594).—Methods for the determination of total benzene hexachloride are reviewed; that of Schechter and Hornstein (*Anal. Chem.*, 1952, **24**, 544) is preferred, although with peanut butter, fats must first be removed. For the determination of the β-isomer, which accumulates in the animal body and is the isomer most resistant to alkaline hydrolysis, a compensatory (compromise) hydrolytic technique (20° C, 3 hr., alcoholic 0·01 *N* alkali) was used. Peanuts planted in soil almost simultaneously with benzene hexachloride application absorb much insecticide, but the ratio of the β-isomer is normal.
A. A. ELDRIDGE

1068. **Determination of iodine in milk.** W. T. Binnerts (*Anal. Chim. Acta*, 1954, **10** [1], 78–80).—After ashing milk at 550° C, the I is extracted with warm slightly alkaline H_2O and determined photometrically by its catalysis of the reaction between $Ce(SO_4)_2$ and arsenous acid. Details of the method are to appear later. J. H. WATON

1069. **A rapid method for the determination of the solubility of powdered milk products.** M. Chilovitch and O. Kornilova (*Ind. Agric. Aliment.*, 1953, **70** [7–8], 567–569].—A sample of dried milk product equivalent to 10 ml of the original liquid is placed in a 10-ml graduated centrifuge tube. It is mixed with 4 to 5 ml of distilled water at 65° to 70° C to a homogeneous paste and the vol. is adjusted to 10 ml. The tube is placed in a water-bath at 65° to 70° C for 5 min., shaken thoroughly and then subjected to centrifugal spinning at 1000 r.p.m. for 5 min. The vol. of sediment should be ≯ 0·2 ml for the best quality and ≯ 0·6 ml for the next quality. This method is in routine use in several laboratories and gives consistent results. N. M. WALLER

1070. **Ash determinations in foods with an alkaline balance. VI. Reaction of sodium carbonate with calcium phosphates in the ashing of milk.** H. J. Wichmann (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 979–992).—The water insol. ash of milk contains Na Ca phosphates and K Ca phosphates. Neutralisation of soured milk with Na_2CO_3 introduces CO_2 (as basic double phosphates or carbonated hydroxyapatite) into the insol. ash. The character of the complex depends on the ratio of alkali to phosphate and the ashing temperature. K is a minor constituent of the water-insol. ash; Cl′ and SO_4'' are present in traces. A. A. ELDRIDGE

1071. **The spectrophotometric identification of dehydracetic acid in cheese.** C. F. Bruening (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 1029).—Dehydracetic acid in 0·1 *N* NaOH gives a max. absorption peak at 292 mμ. The ratio of absorption at 292 mμ to that (in 0·04 *N* HCl) at 307 mμ varies over a narrow range for cheese containing dehydracetic acid. A. A. ELDRIDGE

1072. **[Determination of] dehydracetic acid in cheese.** L. L. Ramsey (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 744–748).—Dehydracetic acid, used as an antimycotic, diffuses into cheese. The quantitative (spectrophotometric) and qualitative methods previously examined (*J. Ass. Off. Agric. Chem.*, 1953, **36**, 83) were subjected to collaborative study. Recoveries of 59 to 103 (average 89·7 per cent. of 100 p.p.m.) are reported. The qualitative test is satisfactory. A. A. ELDRIDGE

1073. **[Determination of] acetic and propionic acid mould inhibitors in bread.** L. H. McRoberts (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 769—781).—The method previously proposed (*Brit. Abstr. C*, 1952, 22) is satisfactory; lactic acid (0·4 to 1·8 per cent.) does not interfere. Butyric acid has not been detected with certainty in milk bread.
A. A. ELDRIDGE

1074. **The meat content of brined and sterilised sausages.** J. F. Reith and M. J. N. Hofsteede (*Analyst*, 1954, **79**, 107–108).—The meat content of Vienna sausages (Frankfurter sausages) pasteurised in brine, calculated by the formula of Stubbs *et al.* (*Analyst*, 1919, **44**, 125) as modified by the Analytical Methods Committee of the Society of Public Analysts and Other Analytical Chemists (*Brit. Abstr. C*, 1953, 85), is invariably lower than that expected from the raw materials used in their manufacture. Analysis of these sausages in various stages of their production shows that decrease of the lean meat content (100 N/3·5) is due to absorption of water from the brine and passage of nitrogenous substances from the sausage into the brine.
A. O. JONES

1075. **[Determination of] total solids and ether extract in fish.** H. M. Risley (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 607–608).—The sample, mixed with sand and Na_2SO_4, is extracted with ethyl ether and light petroleum (1+1) and centrifuged; an aliquot of the extract is evaporated and the residue is dried at 100° C for 15 to 20 min. Preliminary results are satisfactory.
A. A. ELDRIDGE

1076. **[Determination of] solids in raw oysters.** D. C. Price and J. P. Traynor (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 608–616).—If samples are prepared by the Waring blendor method the results (Methods of Analysis, A.O.A.C., Seventh Edition, Sections 18·1 to 18·7) are more accurate than those obtained when the food chopper method is used; moreover, meats and liquids need not be analysed separately. Solids are dried for 3 hr. at 98° to 100° C.
A. A. ELDRIDGE

1077. **The effect of standing, handling and shipping on free liquid, solids and salt content of oysters.** G. T. Daughters and E. M. Hoshall (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 947–954).—The free liquid content of oysters decreased during 1 to 19 days after packing. Values for total solids and salt content are tabulated.
A. A. ELDRIDGE

1078. **[Determination of] lipoids and lipoid P_2O_5 in noodles.** V. E. Munsey (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 760–766).—Collaborative results obtained for egg-yolk noodles were in fair agreement; those for whole-egg noodles were in poor agreement and were usually low. The results are discussed.
A. A. ELDRIDGE

1079. **[Determination of] choline in egg noodles.** V. E. Munsey (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 766–769).—Choline is separated as reineckate and determined photometrically; results for calculated egg content were better for yolk noodles than for whole-egg noodles.
A. A. ELDRIDGE

1080. **Decomposition in tomato products. Determination of acetic, formic, lactic, succinic, malic and citric acids.** H. C. van Dame (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 580–585).—In collaborative work with Bulen, Varner and Burrell's method (*Brit. Abstr. C*, 1952, 271) for acetic, formic, succinic and lactic acids, and a similar chromatographic procedure for citric and malic acids, recoveries of acids added to tomato purée are reported as follows: acetic 68·8 to 100·8 per cent.; formic 43·8 to 103·7 per cent.; lactic 90·2 to 106·0 per cent.; succinic 49·0 to 105·7 per cent.; malic 91·6 to 105·6 per cent.; and citric acid 91·5 to 112·5 per cent.
A. A. ELDRIDGE

1081. **Fill of container studies on frozen fruits.** W. W. Wallace and R. A. Osborn (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 860–871).—The displacement method is satisfactory. Data for commercial packages are recorded.
A. A. ELDRIDGE

1082. **[Determination of] uric acid in fruit products.** D. H. Tilden (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 578–580).—Glacial acetic acid is satisfactory as eluting solvent for the Duolite A-4 (anion-exchange resin) column; if water is added repeatedly during evaporation little caramelisation occurs, and the dry residue, dissolved in saturated Li_2CO_3 soln., gives good resolution of uric acid by paper chromatography.
A. A. ELDRIDGE

1083. **Decomposition in fruit products. [Determination of] galacturonic acid in strawberry juice.** P. A. Mills (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 571–577).—In the determination of galacturonic acid colorimetrically by means of naphtharesorcinol, max. development of colour occurs in 1·25 hr. at 65° C in presence of 0·05 to 0·10 ml of 1 per cent. hydrogen peroxide for a soln. containing 100 μg of galacturonic acid, 20 mg of naphtharesorcinol, 2 ml of aq. acetone (1 + 3) and 2 ml of HCl. Detailed procedure for the construction of a standard absorption curve and for the determination of galacturonic acid in strawberry juice is given. Various varieties of strawberry contained 36 to 56 μg per g of juice.
A. A. ELDRIDGE

1084. **Decomposition in fruits. [Determination of] galacturonic acid, galacturonides and gluconic acid.** W. O. Winkler (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 577–578).—In the carbazole method, whereby higher homologues and polygalacturonides, as well as monogalacturonic acid are determined, "off" colours (not observed with apple juice) are developed by strawberry juice. Degradation of pectin leads to the formation of polygalacturonides containing up to six galacturonic anhydride units. Rotten apples contain significant amounts of gluconic acid.
A. A. ELDRIDGE

1085. **Colour and clarity of gelatin and glue solutions.** P. R. Saunders and A. G. Ward (*J. Sci. Food Agric.*, 1953, **4** [11], 523–527).—The methods normally used do not separately assess colour and clarity of gelatin and glue solutions. By measuring the optical density, either as a function of wavelength with a spectrophotometer or in terms of light transmitted by standard colour filters with a photo-electric absorptiometer, before and after filtering through a Ford Sterimat (GB grade), the colour of the soln. corresponds to the value before filtration and the opacity to the difference the two values. Limitations of the method and its usefulness in routine contol are discussed.
S. C. JOLLY

1086. **[Determination of] gelatin dessert constituents.** Joseph H. Cohen (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 602–605).—Results of collaborative determinations of sucrose and dextrose (Methods of Analysis, A.O.A.C., Seventh Edition, Sections 21·13, 21·14, and 21·15) are assessed statistically. It is now concluded that although for sucrose precision is fairly good, the reproducibility of the results for dextrose is not good.
A. A. ELDRIDGE

1087. **The detection and estimation of parsnip adulteration in prepared horse-radish by infra-red spectrophotometry.** J. Carol and L. L. Ramsey (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 967–969).—When parsnip is used as an adulterant, allyl *iso*-thiocyanate is usually added also. A wide difference in the infra-red spectra of that oil and volatile horse-radish oil appears to offer a method of detection of the adulteration.
A. A. ELDRIDGE

1088. **The detection of algin and gums in cacao products.** F. Y. Mendelsohn (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 599–601).—Locust bean gum, Irish moss or gum arabic can be detected in admixture with reconstituted skim milk, sugar and cocoa by pptn. with ethanol (after treatment with trichloroacetic acid), hydrolysis of the ppt., and testing with Benedict's solution. Algin does not interfere, but no satisfactory test for its presence in such a mixture has been found.
A. A. ELDRIDGE

1089. **[Determination of] chlorogenic acid in coffee.** L. C. Weiss (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 663–670).—Probably owing to destruction of chlorogenic acid during grinding, coarsely ground material is more efficiently extracted than finely ground material; hot water extracts a larger amount of chlorogenic acid than does cold water. The chlorogenic acid content of roasted coffee extracts can be determined by measuring the absorption before and after passage through an anion exchanger, but the Pb pptn. method of Moores, McDermott and Wood (*Anal. Chem.*, 1948, **20**, 620), modified as described, is quicker. A. A. ELDRIDGE

1090. **Separation of uronic and aldobiuronic acids by acid hydrolysis of wheat straw and of certain angiosperm woods.** A. Roudier (*Compt. Rend. Acad. Sci., Paris*, 1953, **237**, 662–663).—Hydrolysis of straw with boiling 9·5 *N* H_2SO_4 followed by neutalisation with $BaCO_3$ and pptn. with ethanol produces a mixture of Ba salts of uronic and aldobiuronic acids; paper chromatography reveals the presence of hexuronic acids and three aldobiuronic acids, which on further hydrolysis with 0·5 *N* HCl are shown to have the following composition: monomethylglucuronic acid-xylose (**I**), glucuronic acid-xylose (**II**), and glucuronic acid-galactose (**III**). **I** and **II** prove to be identical with two acids described by Bishop (*Canad. J. Chem.*, 1953, **31**, 134), and **III** appears to be identical with the aldobiuronic acid from gum arabic. Poplar, lime and aspen wood reveal the presence of a monomethylglucuronic and an aldobiuronic acid corresponding to **I**, but no compound corresponding to **II**, and another compound of unknown composition. These results show that the hemicellulose of wheat straw differs from that of angiosperm wood. It is claimed that the chromatographic separation of acids **I** and **II** had not previously been effected.
J. SCI. FOOD AGRIC. ABSTR.

1091. **Determination of sulphur in barley and malt.** E. Sandegren, D. Ekström and L. Haberl (*Mitt. Versuchssta. Gärungsgew.*, 1953, **7** [9–10], 140–141).—The method of Sandegren *et al.* (*cf. Brit. Abstr. C*, 1953, 222) is improved by packing the combustion tube in the reverse order to that originally given, and by the use of (1 + 2) or (1 + 3) mixture of N and O, instead of a (1 + 1) mixture. The current of gas passing through the tube must be carefully regulated at the outset. The method gives satisfactory results for cystine. Contents of S in Swedish barley (7 samples) are 0·18 to 0·22 per cent., the amount varying inversely with the protein content. Malt contains $\simeq$ 0·9 per cent. of S; during malting, the S is partly absorbed by the embryo and partly ($\simeq$ 50 per cent.) lost in the form of gaseous decomposition products. The barley proteins containing S are probably of low mol. wt. P. S. ARUP

1092. **Change of method for determination of colour of wort and beer.** Analytical Committee of European Brewery Convention (*Mschr. Brauerei, Wissenschaft. Beil.*, 1953, **6** [9], 97–103).—Collaborative tests have shown unsatisfactory results by the Brand method and the iodine method. It has been decided to adopt the E.B.C. standard coloured glasses for colour comparisons. P. S. ARUP

1093. **Measurement of foaming capacity of beer.** P. Kolbach and H. Schilfarth (*Mschr. Brauerei, Wissenschaft. Beil.*, 1953, **6** [6], 61–66).—The measured foaming capacity is not solely a function of the nature of the beer, but varies with the shape and dimensions of the test vessel, the vol. of the sample tested and of the foam layer, temp., and the conditions of foam formation. The setting of the foam on wort follows a quasi-monomolecular course, but no such regularity is observed for the foam on beer. An apparatus is described which consists of a 600-ml cylinder, 70 mm in diameter, furnished with a glass tap at the bottom (22 cm from the top); for the dispersion of the CO_2, a Jena f 33 CG 2 dispersion tube is used. Determinations should be made under standard conditions in apparatus calibrated with a liquid of standard foaming capacity. P. S. ARUP

1094. **Determination of yeast-fermentable sugar in beer.** A. P. Mathers and J. E. Beck (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 954–957).—Procedure for determination (by means of dichromate or ceric salt) of the alcohol produced by yeast fermentation of de-alcoholised beer is described.
A. A. ELDRIDGE

1095. **Chromatographic determination of glycerol in fermentation solutions.** K. Sporek and A. F. Williams (*Analyst*, 1954, **79**, 63–69).—A chromatographic procedure is described for the rapid determination of glycerol in mixtures with sugars and the constituents of molasses. The adsorbent is Al_2O_3 resting on a cellulose column and the solvent is acetone containing 5 per cent. v/v of water and 0·05 per cent. of glacial acetic acid. To assist retention of sugars on the column, Na_2SO_3 and Na acetate are added to the sample soln. Glycerol is determined in the eluate, after removal of the solvent, by titration of the formic acid produced by oxidation with $NaIO_4$ (Erskine *et al.*, *Anal. Abstr.*, 1954, **1**, 498). A. O. JONES

1096. **Paper chromatography in the qualitative and quantitative analysis of organic flavouring compounds.** R. ter Heide and J. F. Lemmens (*Perf. Essent. Oil Record*, 1954, **45** [1], 21–23).—Vanillin, ethylvanillin, methylvanillin, heliotropin, vanitrope and coumarin, which are the normal constituents of vanilla flavours, can be separated by paper chromatography with a developer comprising benzene - light petroleum (80° to 110° C) - methanol in a vol. ratio of 2 to 10 to 1, in a neutral medium. The R_F values for three types of filter-paper are tabulated and the spot-test reagents are described. For quantitative analysis, 5 μl (corresponding with 5 μg of 0·1 per cent. standard soln. of the mixture in 96 per cent. ethanol, is chromatographed and developed. The zones are cut out and extracted with 96 per cent. ethanol in a micro Soxhlet apparatus, the extracts being made up to a definite vol.; the quantitative measurement is made spectroscopically. The concn., max. extinction and wavelengths of the various compounds are given. Blank extractions are essential. A complete analysis takes about 8 hr. G. HELMS

1097. **[Analysis of] vanilla extracts and imitations. A. Determination of vanillin, ethylvanillin and coumarin by ultra-violet absorption. B. Colorimetric determination of vanillin and coumarin.** L. G. Ensminger (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 679–697).—Absorptivities for vanillin, ethylvanillin and coumarin at 270, 348, 247 and 309 mμ are recorded. The ultra-violet method afforded good results for vanillin and vanillin plus ethylvanillin, the average recovery in collaborative work being 95·6 per cent. For coumarin, present

in small amount, the average recovery was 98·2 per cent., although the precision was slightly less than that of the colorimetric method. In nearly all the extracts examined the authentic and imitation vanilla extracts could be differentiated as the former give average absorptions at 247 mμ (acidic) and 270 mμ (basic) of more than 0·020, whilst the latter give lower values. Vanillin and coumarin in an enriched vanilla extract and in an imitation vanilla flavouring were satisfactorily determined by the colorimetric method, recoveries of both being in the range 97·6 to 102·1 per cent.

A. A. ELDRIDGE

1098. **Spectrophotometric detection of certain types of adulterants in vanilla.** R. M. Roberts (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 958–967).—The sample is defecated with Pb acetate, and vanillin and any other phenolic substances and aromatic carboxylic acids are removed with NaOH. The residue obtained on evaporation of the ethereal extract is dissolved in acidified ethanol and its absorption is measured. Absorption curves for various commercial samples and possible adulterants are given.

A. A. ELDRIDGE

1099. **[Detection and determination of] artificial sweeteners. P-4000 (propoxy-2-amino-4-nitrobenzene).** W. S. Cox (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 749–750).—The substance, extracted by means of light petroleum, is reduced with $SnCl_2$ and the product is brominated, giving a burgundy-coloured soln. Gialdi's method of determination (*Farm. Sci. Tec.*, 1948, **3**, 44) by diazotisation and coupling with 1-naphthol is modified; the colour is extracted with *iso*pentanol, the soln. having an absorption max. at 515 mμ.

A. A. ELDRIDGE

1100. **[Determination of] volatile oil in spices.** N. A. Carson (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 752–757).—Apparatus having glass joints should be used. Average values for volatile oil (per cent. v/w), n_D^{20} and sp. gr. (25°/25°) were, respectively: star anise 6·64, 1·5528, 0·9763; nutmeg 7·47, 1·4879, 0·9215; and allspice 4·12, 1·5285, 1·0279 (eugenol, 80·1 per cent. of oil).

A. A. ELDRIDGE

1101. **Analysis of liquids, particularly fatty oils and salt solutions, with the aid of micro-determination of the critical mixing temperature.** R. Fischer and J. Horner (*Mikrochim. Acta*, 1953, [4], 386–400).—In the usual rapid micro-determination of critical mixing temp. the value obtained is always that corresponding to the max. on the miscibility diagram. The critical mixing temp. was determined for several fatty oils with ethylene chlorohydrin. Oils from different sources gave the same results. Standard curves for mixtures of oils with oleic acid and a paraffin oil have been obtained with ethylene chlorohydrin. Mixtures of gourd-seed oil with rape-seed oil and of paraffin oil or chloroform with fatty oils can also be tested in the same way. The critical mixing temp. can also be used to determine the concn. of aq. solutions of inorganic salts and some organic compounds. Six to eight concn. of each were used to determine the critical mixing temp. with phenol as test liquid and standard curves can be obtained. A determination takes 10 to 15 min. and the accuracy is 0·3 per cent. per degree of temp. difference. There is a complete miscibility gap for α-picoline with dil. aq. NaCl.

A. J. MEE

1102. **Separation of sterols by adsorption.** M. S. Muñoz (*An. Bromatologia*, 1953, **5** [3], 307–315).—The separation of sterols from the rest of the components of the unsaponifiable matter is difficult as many natural fats contain pigments and fluorescent compounds, which interfere with the isolation of sterols by normal means. By chromatography, however, it is possible to effect this separation and hence distinguish between fats of different origins.

The chromatographic behaviour of various sterols representative of the animal and vegetable kingdoms was compared with the aid of two types of active alumina. The sterols were tested singly and in mixtures of the pure sterols and their esters. It was found possible to separate the various components of mixtures and reveal their position on the column with u.v. light, but elution proved difficult and often they were so strongly fixed to the adsorbent that they could not be removed at all. This applies particularly to phytosterol.

H. PRITCHARD

1103. **Use of 1:2-dichloroethane in the Carr-Price antimony trichloride reagent for the determination of vitamin A.** A. B. McCoord (*J. Lab. Clin. Med.*, 1953, **42** [4], 660).—Chloroform used as a solvent for antimony trichloride in the Carr - Price reagent requires careful purification before use. 1:2-Dichloroethane can be used without such purification. The reagent is prepared by dissolving 30 g of $SbCl_3$ in 100 ml of 1:2-dichloroethane. The intensity of colour produced with vitamin A is equal that produced with the Carr - Price reagent. Corrections for carotene and xanthophyll must be made and these are determined by the method of Clausen and McCoord (*J. Biol. Chem.*, 1936, **113**, 89). The colour produced by the reagent with 100 μg of carotene is equal to that produced by 17 U.S.P. units of vitamin A, and that produced with 100 μg of xanthophyll equal to that produced by 22 U.S.P. units.

D. C. M. ADAMSON

1104. **[Determination of] vitamin A in margarine. Blank-oil method compared with the chromatographic procedure.** J. B. Wilkie (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 820–837).—Considerations affecting the accuracy of the determination are reviewed. Tabulated values indicate that the blank oil technique and $SbCl_3$ before chromatography give high values, probably owing to irrelevant absorption that should be obviated chromatographically. A method involving chromatography, based on that of Wilkie and De Witt (*Brit. Abstr. C*, 1945, 184) and described in detail, was used for collaborative study. Tentative conclusions concerning the significance of the results are reached.

A. A. ELDRIDGE

1105. **[Determination of] vitamin A in mixed feeds.** M. L. Cooley (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 812–819).—Collaborative results are considered statistically. The method used (*Brit. Abstr. C*, 1953, 272) appeared to be satisfactory for determining ≯ 1 p.p.m. of vitamin A, but as saponification is omitted, lipids and other extracted materials interfere. A procedure, including saponification, for stabilised vitamin-A products is proposed.

A. A. ELDRIDGE

1106. **The determination of thiamine in enriched flour. Comparison of acid hydrolysis and fluorimetric methods.** L. H. McRoberts (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 837–845).—Variations in the rapid procedure, involving acid hydrolysis

and measurement of thiochrome fluorescence, were subjected to collaborative study, and the results are tabulated. Either the "determination" or the "direct" standard is applicable, and either centrifuging or filtration can be applied in preparing the sample solution. A. A. ELDRIDGE

1107. **The use of ion-exchange resin in the quantitative chemical differentiation between nicotinic acid and nicotinamide.** J. P. Sweeney and L. Hall (*J. Ass. Off. Agric Chem.*, 1953, **36** [3], 1018–1022).—The sample soln., at pH 5, is passed over the anion-exchange resin IRA-400. The nicotinamide is eluted with water, and the nicotinic acid with hot *N* HCl. Both substances are then determined by means of König's reaction with CNBr and sulphanilic acid (*Brit. Abstr. C*, 1952, 21).
A. A. ELDRIDGE

1108. **Differential determination of vitamin-B_6 groups.** S. Fukui (*Anal. Chem.*, 1953, **25** [12], 1884–1886).—By application of cation-exchange resins, vitamin-B_6 extracts are separated into two fractions, one of which contains pyridoxamine, the other pyridoxal and pyridoxine. The vitamin content of the fractions is determined by microbiological assay by means of *Saccharomyces carlsbergensis* 4228. The pyridoxine is determined after destruction of pyridoxal by treatment of a separate fraction with acetone and NaOH. The resins used are KH4B (synthesised by condensation polymerisation of phenoxyacetic acid and formaldehyde) and Duolite C60. The recoveries are between 95 and 105 per cent. G. P. COOK

1109. **Application of paper chromatography for the determination of vitamin B_{12} in liver extracts.** J. Bogucka, J. Iwanowska and H. Kakol (*Przem. Chem.*, 1953, **32** [10], 512–413).—A biological method for the determination of up to 1 μg per ml of vitamin B_{12} in liver extracts has been described by the authors (*Przem. Chem.*, 1953, **32**, 14). Now a paper chromatographic method is reported, which allows the determination of a wide range of vitamin-B_{12} contents in liver extracts of various degrees of purity, with the aid of an apparatus described by Hermanowicz and Obuchowska (*Przem. Chem.*, 1950, **29**, 649). Sample of 0·002 ml of the examined soln. are placed on paper strips previously impregnated with KH_2PO_4 and dried at 40° C. A mixture of 8 ml of *n*-butanol, 10 ml of ethanol and 100 ml of water serves as solvent. The dry chromatogram is developed on agar inoculated with a strain of *E. coli*. After 18 hr. a well-defined elliptic chromatogram is obtained, which is measured planimetrically. The vitamin-B_{12} content is calculated with the aid of a graph, in which for a standard vitamin-B_{12} soln. the chromatographic zones are plotted against the log. of vitamin B_{12} concn. H. BURSTIN

1110. **Spectrophometric method for the determination of vitamin B_{12} in microbiological cultures.** J. Janicki, J. Pawelkiewicz, S. Stawicki and K. Zodrow (*Przem. Chem.*, 1953, **32** [10], 509–511).—According to this method which is a modification of Rudkin and Taylor's procedure (*Brit. Abstr. C*, 1952, 562), the dil. vitamin soln., obtained by extraction of the microbiological culture with benzyl alcohol, is washed with water and extracted from this with acetone after saturation with $(NH_4)_2SO_4$. The vitamin B_{12} is obtained in conc. form by evaporation of the acetone under reduced pressure. The vitamin content is determined by comparing the extinction at 588 mμ of the dicyanate complex with that of a standard vitamin soln. The extinction coeff. established was $E^{1\%}_{1\,cm} = 58{\cdot}2$. The deviation from the Rudkin - Taylor extinction coeff., $E^{1\%}_{1\,cm} = 54$ at 582 mμ, is attributed to use of different optical systems. The spectrophotometric method (reproducibility ± 10 per cent.) can be applied to all microbiological preparations. Fair agreement was established with results of microbiological tests with *Euglena gracilis*.
H. BURSTIN

1111. **[Determination of] vitamin B_{12} (microbiological methods).** C. H. Krieger (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 846–856).—In the method described and used for collaborative study, buffered $NaHSO_3$ was used to stabilise vitamin B_{12b} and the growth of the *Lactobacillus leichmannii* was evaluated turbidimetrically after incubation for 16 to 24 hr. The coeff. of variation of the results for four samples varied from 10·8 to 50·6 per cent., being highest for a sample of low potency (evaporated milk). A. A. ELDRIDGE

1112. **The effect of ascorbic acid and trace elements on vitamin-B_{12} assays.** E. M. Stapert, E. B. Fevrer and L. Stubberfield (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [2], 87–90).—The stability of vitamin B_{12} in soln. containing ascorbic acid is influenced by $Cu^{\cdot\cdot}$, MoO_3'', F' and $Mn^{\cdot\cdot}$, which caused destruction of vitamin B_{12} while $Co^{\cdot\cdot}$, $Mg^{\cdot\cdot}$, I', $Fe^{\cdot\cdot}$ and $Zn^{\cdot\cdot}$ did not. Copper was the most active. None of these ions when tested separately affected the stability of vitamin B_{12} in soln. N. E.

See also Abstracts 967, 987, 988.

Sanitation

1113. **Apparatus for determining the condition of water as regards its content of predetermined types of impurities.** Filtrators, Ltd., and E. L. Streatfield (*Brit. Pat.* 695,638, 13.4.49 and 12.8.49).—Water taken continuously or at intervals from a sampling point is delivered to two mixing chambers. In one the water is untreated. In the other it is mixed with a measured quantity of a reagent that reacts with the predetermined impurities to cause pptn. or colour change. Light is transmitted through both chambers on to two photo-electric cells. The cells react according to variations in the light falling on them. Apparatus is connected to the cells, and the whole photo-electric apparatus is unaffected by changes in the water other than those caused by pptn. or colour changes caused by reaction between the added agent and the predetermined types of impurities in the water. In one form of apparatus the water flows continuously through two transmission cells (each with an inlet and an outlet adjacent to its ends); transparent ends enable light to be transmitted to the appropriate photo-cell after passing through the treated water and the "control." In another form of apparatus the light passing through one cell is varied relatively to that passing through the other until the electromotive forces generated by the cells balance.
J. SCI. FOOD AGRIC. ABSTR.

1114. **Determination of total hardness of boiler waters by a photocolorimetic method.** J. E. Goris and J. L. de Hauss. (*Chim. et Ind.*, 1953, **70**, 420–424).—A method based on measurements of the optical density of samples in which turbidity has been produced by addition of soap soln. is described. By following a standardised procedure good reprodicibility and an accuracy of 0·1° of hardness are attained. J. SCI. FOOD AGRIC. ABSTR.

1115. **Evaluation of the oxygen-consumed test.** Hazel V. Roberts and W. W. Sanderson (*Sewage & Ind. Wastes*, 1953, **25** [7], 793–797).—The work of Moore *et al.* (*Anal. Chem.*, 1951, **23**, 1297) on the comparison of various types of oxygen-consumed tests is discussed. Moore found that the $K_2Cr_2O_7$ method developed by the U.S. Public Health Service is the most satisfactory of the methods proposed. In general, branched-chain hydrocarbons, sugars and phenolic compounds are readily oxidised, but straight-chain acids and alcohols and compounds such as benzene, pyridine and toluene are not. It was found later, however, that addition of 1 g of Ag_2SO_4 to the sample before refluxing increased markedly the oxidisability of the straight-chain acids and alcohols, *e.g.*, acetic acid (which otherwise is scarcely attacked) is oxidised to 95 per cent. of the theoretical, while ethanol and lactic acid are broken down to a considerable extent (> 80 per cent.). Oxidation of benzene, pyridine and toluene is unaffected by addition of Ag_2SO_4. However, Ag_2SO_4 is incompatible with some samples, particularly those with a high Cl′ content, and should only be used on wastes that require it. The method is unsuitable for the analysis of samples that have low oxygen-consumed values, such as those from unpolluted streams. J. M. JACOBS

1116. **Coliform detection in water by a single-step technique using the membrane filter.** A. A. Hajna and S. R. Damon (*Publ. Hlth. Rep., Wash.*, 1954, **69** [1], 58–60).—A single-step procedure for coliform detection in water analysis by the millipore filter is described. The formula for a deoxycholate - lactose broth to be used in the millipore-filter technique is given. With the deoxycholate - lactose-broth medium in the millipore-filter procedure for water analysis enrichment of the filter is eliminated. Results are obtained earlier with this technique than with standard methods of water analysis. Many of the false positive lactose-broth tests encountered in standard methods of water analysis are eliminated. I. JONES

1117. **Determination of pyrethrin I.** V. A. Beckley and J. Hopkins (*Soap*, 1954, **30** [1], 141–147 and 173).—A study of alternative techniques for assaying pyrethrin I by the mercury-reduction method shows that the official technique of the A.O.A.C., which involves filtration after acidification with H_2SO_4 in order to remove $BaSO_4$, yields low results. These are not due to occlusion of chrysanthemic acid by the $BaSO_4$, but to occlusion in a flocculent precipitate of water-insoluble acids. G. HELMS

See also Abstracts 935, 985.

Agriculture and Plant Biochemistry

1118. **Procedures for the extraction, separation and estimation of the major fat-soluble pigments of hay.** J. Davidson (*J. Sci. Food Agric.*, 1954, **5** (1), 1–7).—New methods are described for the extraction, chromatographic separation and spectrophotographic identification and determination of chlorophylls *a* and *b*, phaeophytins *a* and *b*, carotene (**I**) and xanthophyll (**II**) in hay. Chlorophylls in the extract were separated from the other pigments on sucrose - Na_2SO_4 mixture and were determined simultaneously by measuring the light absorption of the green fraction at 661 and 642·5 mμ; the phaeophytins were determined simultaneously by measuring the absorption at 655 and 667 mμ. Subsequently **I** and **II** were separated by chromatographing the yellow fraction on MgO and were determined individually by measuring absorption of the eluates at 450 and 444 mμ, respectively. S. C. JOLLY

1119. **Qualitative paper chromatography of sugars in plants.** K. T. Williams and A. Bevenue (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 969–979).—Convenient apparatus and procedures are reviewed in detail and the interpretation of the chromatogram is discussed. A. A. ELDRIDGE

1120. **Paper chromatography of chlorophylls.** A. H. Sporer, S. Freed and K. M. Sancier (*Science*, 1954, **119**, 68–69).—Impregnation of the paper with sucrose prevented the decomposition previously experienced with chlorophylls. The developer used was 0·5 per cent. *n*-propanol in *n*-hexane. Chlorophylls *a* and *b* were separated into two distinct spots; likewise chlorophylls *b* and *b*′. N. E.

1121. **[Determination of] carotene.** F. W. Quackenbush (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 857–860).—Variations in results obtained cannot be attributed mainly to deterioration, between preparation and analysis of the samples of lucerne. A. A. ELDRIDGE

1122. **Determination of α-amino-nitrogen complexes in soils.** W. Laatsch and E. Schlichting (*Z. PflErnähr. Düng.*, 1953, **62**, 50–63).—Possible causes of loss of α-amino-nitrogen are investigated. By using as test-substances compounds of gelatin with synthetic humic acid, tannin, catechol and lignin, complete hydrolysis resulted after boiling for 12 hr. with 6 *N* HCl. Experiments with added alanine revealed losses of α-amino-nitrogen on boiling with 6 *N* HCl in presence of soils containing MnO_2; the losses increased with the degree of subdivision of the MnO_2 mineral, and decreased with the degree to which it had been weathered. J. SCI. FOOD AGRIC. ABSTR.

1123. **Determination of zinc [in soils] with special reference to separation from cobalt and copper.** A. E. Martin (*Anal. Chem.*, 1953, **25** [12], 1853–1858).—The extraction of Zn from citrate solutions at pH 5 to 10 with di-2-naphthylthiocarbazone (**I**) and Na diethyldithiocarbamate was investigated and a tentative method for Zn in the presence of Cu and Co is suggested. The sample soln. or digest containing 2 ml of conc. H_2SO_4 is diluted to 50 ml and is mixed with 50 ml of 0·096 *M* ammonium citrate (pH 10·8). This soln. is extracted with 5 ml of soln. of **I** (250 mg per litre of $CHCl_3$) and the Zn in the solvent phase is extracted into 20 ml of 0·5 *N* HCl. The acid extract is evaporated to dryness and the organic matter is destroyed by treatment first with dil. HNO_3 and then with 2 drops of conc. H_2SO_4 and 0·5 ml of 72 per cent. $HClO_4$. The soln. is evaporated to dryness, ignited at 300° to 350° C and the Zn is determined by polarographic analysis on this residue, the base soln. used being 0·1 *M* in NH_4Cl, 0·02 *M* in KCNS and containing 0·0002 per cent. of methyl red. The diffusion current is measured between − 0·8 and − 1·3 V *vs.* the S.C.E. The mean recovery of Zn is 98·8 per cent. over the range 10 to 300 μg, the standard error being ± 0·8 per cent. Satisfactory recoveries are also obtained in the presence of Cu, Co, Fe, PO_4''' and Ti at the concn. likely to be encountered in soils. G. P. COOK

1124. **Purification of activated charcoal for decolorising soil extracts.** S. H. Yuen and A. G. Pollard (*J. Sci. Food Agric.*, 1953, **4** [11], 503–507).—Nearly all the phosphate extractable by the usual soil extractants is removed from B.D.H. activated charcoal by boiling twice with 10 vol. of HCl (sp. gr. 1·18) for 3 hr., and then washing with NaCl soln. The purified charcoal used at the rate of $\geqslant$ 0·1 g per 25 ml of soln. decolorises 2·5 per cent. acetic acid extracts and Morgan extracts of ordinary field soils, but does not completely decolorise 1 per cent. citric acid extracts. NH_3, NO_3', PO_4''', K, Ca, Mg and Mn are adsorbed to varying extents from aq. solutions; from acid solution (0·002 *N* H_2SO_4) adsorption of NO_3' was further intensified, but that of the other ions is insignificant. S. C. JOLLY

1125. **Determination of maleic hydrazide residues in plant and animal tissue.** P. R. Wood (*Anal. Chem.*, 1953, **25** [12], 1879–1883).—The maleic hydrazide is reduced and hydrolysed to hydrazine by heating with NaOH soln. and Zn in a distillation apparatus, the distilled hydrazine being collected in H_2SO_4. The hydrazine is then determined colorimetrically at 455 mμ with the aid of *p*-dimethylaminobenzaldehyde. Interfering substances are pyrrole, pyrogallol, resorcinol, pyrazoles, piperidine and compounds that break down to SO_2. Samples containing $<$ 1 μg of maleic hydrazide can be assayed and duplicates on various plant samples agree to within $\pm$ 10 per cent. G. P. COOK

1126. **X-ray examination for the detection of internal insect infestation in corn.** J. F. Nicholson, O. L. Kurtz and K. L. Harris (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 993–1001).—Shadows produced on the radiographs of maize kernels by normal structures can be differentiated from those produced by insect damage. The gross damage so assessed agrees with that determined by the cracking flotation method. A. A. ELDRIDGE

1127. **A new flotation method for the determination of insect infested wheat.** D. B. Scott (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 1026–1027).—The kernels are coated to mol. thickness with a volatile silicone before flotation in 20 per cent. NaCl soln. The procedure is more efficient for separation of infested and undamaged kernels than when silicone treatment is omitted. A. A. ELDRIDGE

1128. **Sampling and preparation of fertiliser sample.** S. B. Randle (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 617–622).—The sample was quartered in the field, and both large and small fractions were analysed collaboratively for N, K_2O, total P_2O_5 and available P_2O_5. Tabulated results indicate that there are variations in analysis of the samples quartered in the field. A. A. ELDRIDGE

1129. **Use of flame photometry to determine potassium [in fertilisers] at 404 millimicrons.** A. T. Blackwell, C. L. Yeager and M. Kraus (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 898–902).—Determination of K with a Beckman model DU spectrophotometer affords results as precise and accurate as obtained by the A.O.A.C. procedure and more rapidly. A. A. ELDRIDGE

1130. **Determination of potassium in mixed fertilisers.** R. C. Crooks (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 891–898).—The calibration of the Beckman Model DU flame photometer is discussed, and procedure for its use is described. It provides a rapid and accurate method for screening mixed fertilisers for K content. Dil. solutions (max. 50 p.p.m. as K_2O) prepared by the A.O.A.C. procedure, a wavelength of 767 mμ and a narrow slit are used. A. A. ELDRIDGE

1131. **Potassium analysis of twelve years' Magruder check samples [of fertilisers] by flame photometry.** E. D. Schall (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 902–907).—The samples (containing 2·0 to 62·5 per cent. of K) were analysed by the use of a Beckman model B spectrophotometer with flame attachment. The results were generally slightly lower than the Magruder values (standard deviation, $\pm$ 0·2 per cent.). Phosphate and oxalate increased the apparent K content, and these ions were therefore added to the standard soln. in compensation. A. A. ELDRIDGE

1132. **[Determination of] potash [in fertilisers].** O. W. Ford (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 649–654).—Collaborative results show that whilst the flame-photometric method gives for pure KCl results identical with those afforded by the A.O.A.C. and modified Perrin methods, for mixed fertilisers the results are slightly higher. A. A. ELDRIDGE

1133. **[Determination of] boron in mixed fertilisers.** R. C. Berry (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 623–628).—In a modified method for the determination of water-sol. B, $BaCl_2$ is used as precipitant, after neutralisation with $Ba(OH)_2$ to remove PO_4''', SO_4'', and CO_3''. Collaborative results for for 3 mixed fertilisers are tabulated. The modified method is less troublesome; results were fairly accurate. A. A. ELDRIDGE

1134. **[Determination of] magnesium in fertilisers.** John B. Smith and C. E. Olney (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 628–632).—Collaborative results for the determination of Mg in particles of fertiliser retained by a 40-mesh sieve (openings 420 μ)—the fraction of little immediate value as a fertiliser—are reported. The sample is repeatedly disintegrated in water before being poured through the sieve. A. A. ELDRIDGE

1135. **[Determination of] inert materials in fertilisers: carbonate carbon or calcium carbonate equivalent and acid-insoluble ash.** K. G. Clark and V. L. Gaddy (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 655–661).—Collaborative results are reported and statistically significant differences are discussed. The procedures detailed need modification. A. A. ELDRIDGE

1136. **[Determination of] nitrogen in fertilisers.** H. A. Davis (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 644–649).—Tabulated collaborative results obtained by the A.O.A.C. method (Methods of Analysis, 1950, 2·25 or 2·26) and Shuey's method (*Brit. Abstr. C*, 1951, 186) indicate that the former is to be preferred for general use. A. A. ELDRIDGE

1137. **Chemical method for [determining] available fertiliser nitrogen in urea-formaldehyde compositions.** W. A. Morgan and R. D. Kralovec (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 907–914).—The availability index, which can be used for predicting agronomic value, is defined as the percentage of the cold-water insol. N (determined by the A.O.A.C. method) that dissolves in a hot aq. phosphate buffer soln. (determined on another sample). Procedure is described. A. A. ELDRIDGE

1138. **Direct determination of available phosphoric acid in fertilisers.** H. R. Allen (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 872–874).—Phosphorus in the combined water- and citrate-soluble extracts is pptd. as ammonium molybdophosphate with continuous agitation for 1 hr. at 50° C. Addition of NH_4NO_3 is unnecessary. The method is applicable to mixed fertilisers and superphosphate but not to calcium metaphosphate. A. A. ELDRIDGE

1139. **Direct determination of available phosphoric acid [in fertilisers] by volumetric and photometric procedures.** K. D. Jacob, W. M. Hoffman and F. C. Schramm (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 632–644).—The official (A.O.A.C.) procedure afforded the best precision, and the results were somewhat higher than those obtained by the direct methods. The direct photometric method afforded the poorest precision. Direct volumetric determination of available P_2O_5 in superphosphates and mixed fertilisers gave lower results if extraction with water was omitted than when water was used. A. A. ELDRIDGE

1140. **A modified spectrophotometric procedure for the direct determination of available phosphoric acid in fertilisers as the heteropolyphosphovanadomolybdate complex.** R. T. Teague, jun. (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 880–885).—Phosphorus in the combined water- and citrate-soluble extracts is converted into PO_4''', which is determined spectrophotometrically (Epps, *Brit. Abstr. C*, 1951, 61; Kitson and Mellon, *Brit. Abstr. C*, 1944, 158). Sulphate does not interfere. The method is applicable to fertilisers containing organic phosphates and metaphosphates. A. A. ELDRIDGE

1141. **Instrumental methods for the determination of available phosphoric acid and potash in fertilisers.** H. C. Austin, jun., W. P. Denson and E. A. Epps, jun. (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 885–890).—Epps' photometric method for determining available PO_4''' (*Brit. Abstr. C*, 1951, 61) is rapid and accurate. Except with muriate of potash, the flame photometric method for determining K is also rapid and as accurate as the A.O.A.C. method. Both determinations may be made on one soln. Procedure is detailed. A. A. ELDRIDGE

1142. **The use of perchloric - nitric acid digestion in the determination of phosphoric acid in fertilisers.** L. J. Hardin (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 874–879).—Accurate results for the P_2O_5 content of mineral and organic materials are obtained by digestion with $HClO_4$ - HNO_3 followed by volumetric determination. Much SO_4'', if present, causes high values, so that the usual volumetric procedure is inapplicable after digestion with H_2SO_4 - HNO_3; digestions with HNO_3 - HCl do not extract all the organically combined P. A. A. ELDRIDGE

1143. **A constant-temperature water-bath for use in determination of available phosphoric acid in fertilisers.** J. A. Shrader and H. R. Allen (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 1023–1024).—The bath is heated electrically and is provided with a Fenwal thermoregulator and with a flask-supporting shaft, which is given a reciprocating and rocking motion. A. A. ELDRIDGE

1144. **A large-capacity constant-agitation bath for use in citrate-insoluble phosphoric acid determinations.** W. P. Matthews (*J. Ass. Off. Agric. Chem.*, 1953, **37** [3], 1024–1026).—The bath is electrically heated and is provided with a Fenwal thermostat. The flasks are held in cups on a platform which is caused to move laterally on ball bearings. A. A. ELDRIDGE

See also Abstracts 894, 910, 1067, 1091, 1105.

5—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

1145. **A humidity-control system for air-conditioned cabinets.** F. J. F. Fisher (*N. Z. J. Sci. Tech., B*, 1953, **35** [3], 279–283).—Details of the construction of two controlled-environment cabinets are given. By using a refrigeration system more powerful than is normally used for temperature control alone, a wide range of humidity control is achieved (by indirect thermostatic control) at little cost. Data relating to performance are: amplitude of temp. ± 1·4° C; amplitude of humidity ± 4 per cent. R.H.; max. temp. 38° C and min. −6° C; temp.-cycle period at high R.H. 30 min. and at low R.H. 4 min.; max. humidity 100 per cent. R.H., min. humidity 22 per cent.; min. humidity at 28° C, 74 per cent. R.H.; humidity cycle at high R.H. 90 min. and at low R.H. 4 min. G. HELMS

1146. **A trough for paper chromatography consisting of segments.** G. Wunderly (*Nature*, 1954, **173**, 267–268).—To facilitate measurements under identical conditions in descending paper chromatography, a glass trough is made up of four equal segments, so enabling the use of duplicate papers (7 × 40 cm) or two sheets (slotted) 40 × 40 cm. Experimental results show that a pH gradient can be established across the paper with buffers of pH differing little in successive segments of the trough; hence a more complete separation of protein hydrolysate fractions containing polar groups is effected. This technique is applied to protein hydrolysates after first being used with propanol - water (7 + 3). D. E. BLENFORD

1147. **Device for rapid measurement of chromatographic R_b [R_F] values.** A. J. Glazko and W. A. Dill (*Anal. Chem.*, 1953, **25** [11], 1782.—A proportional divider for measuring chromatographic R_F values is described, the measurements being as accurate as those attained by manual measurements with a ruler; the R_F values are read off directly from the end of a pointer. G. P. COOK

1148. **A modified liquid - liquid extraction apparatus.** K. W. Mieszkis (*Analyst*, 1954, **79**, 109–110).—A disadvantage of the liquid - liquid extraction apparatus of Clasper *et al.* (*Brit. Abstr. C*, 1950, 15) is indicated and a modification consisting in a combination of a Soxhlet extractor and the above-mentioned apparatus free from the disadvantages is described. A. O. JONES

1149. **Improved glass plug for filtration.** B. Flaschentträger and S. M. Abdel-Wahhab (*Mikrochim. Acta*, 1954, [1], 72–73).—The method of constructing the plug from a Pyrex-glass rod is described. A. J. MEE

1150. A cyclic laboratory evaporator. G. Ducellier (*Chim. Anal.*, 1954, **36** [1], 18–19).—An apparatus is described which can be used to concentrate 1 to 2 litres of liquid to one-tenth of its volume without overheating thermolabile substances in soln. The main heating surface is so arranged that the liquid is continually circulated and spreads in a thin layer on the walls of the concentrator. E. HAYES

1151. A micro-molecular still. R. F. Paschke, J. R. Kerns and D. H. Wheeler (*J. Amer. Oil Chem. Soc.*, 1954, **31** [1], 5–7).—The body of the still consists of 3-cm bore glass tubing, the upper part of which houses a spring-balance device consisting of a 60-turn quartz helix (sensitivity 2·92 cm per g) carrying a crosswire, and a scale. The sample ($\simeq$ 0·5 g) is distributed on a roll of sheet glass-wool, carried by a wire holder, which is suspended from the quartz helix by means of a glass fibre (hooked at both ends). The part of the tube enclosing the sample and its carrier (connected with the upper part by a ground-glass joint) is surrounded by an aluminium-block heater (removable), which carries thermometers and an adjustable thermoregulator. At 2 to 3 cm below the heater the tube is constricted (14/35 ground-glass joint) to take either a device for collecting distilled fractions in ampoules or a simple "dry-ice" cooled trap. The part of the tube below the heater is cooled by a jet of air. In the ampoule model, the passage by way of a side-tube to the mercury-diffusion pump (which, backed by a mechanical pump, reduces the pressure to 1 to 5 μ) is intercepted by a small "dry-ice" trap. Suitable heating temp. for distillation of the various (monomeric, dimeric or trimeric) fractions of polymerised products are determined experimentally. In the fractionation of normal polymers, *e.g.*, in following the rate of polymerisation of the methyl elaeostearates, or in following the kinetics of the dimerisation of methyl linoleate by di-*tert.*-butyl peroxide, the new method is preferable to existing methods on account of its suitability for working with small samples with min. heat damage. P. S. ARUP

1152. Brittingham laboratory pump. L. R. Setter, W. E. Brittingham and R. F. Wessels (*Sewage & Ind. Wastes*, 1953, 25 [7], 798–801).—The Brittingham squeegee pump is described in which a flexible stretched tube is rolled over by three or four rollers mounted on a disc rotating at 37 r.p.m. The pump is cheap, non-clogging and self-priming. The thermal effects are negligible even when pumping 3 per cent. sewage sludge. The flexible tube (*e.g.*, of amber rubber), which acts as a piston, lasts for several months. Examples of its operating characteristics are given. J. M. JACOBS

1153. A reciprocating mercury pump for gas analysis apparatus. E. A. C. Chamberlain, S. R. M. Dixon and P. L. Waters (*J. Sci. Instrum.*, 1954, **31** [2], 66-67).—The pump described automatically raises and lowers the mercury level in a Haldane-type gas-analysis apparatus. G. SKIRROW

1154. An improved accurate film balance. K. Inokuchi (*Bull. Chem. Soc. Japan.*, 1953, **26** [8], 471–475).—A new apparatus of float-balance type for measuring low film pressures in the study of monomolecular layers is described and its mode of operation is detailed. F. R. MUMFORD

1155. Laboratory apparatus for testing the chemical resistance of plastics. M. Grochowski (*Przem. Chem.*, 1953, **32** [8], 404–405).—Two round-bottomed 250-ml hard-glass bottles are each equipped with reflux condenser and short side-tube (25 mm int. diam.). The ends of the side-tubes have ground flanges which fit to each other. The disc-shaped plastic to be tested is placed between the two flanges and these are tightly fixed to each other by a double metal ring with bolts and nuts. One bottle is filled with the corrosive liquid and the other one with an inert fluid (*e.g.*, HCl and water). The bottles are heated to testing temp. and the time required for the corroding liquid to penetrate through the plastic disc into the inert fluid is recorded. The critical point is indicated by a suitable reagent, *e.g.*, phenolphthalein. Microscopic inspection supplements the test. H. BURSTIN

1156. An instrument for determining the relaxation and recovery of elastomers. B. G. Labbe and W. E. Phillips (*India Rubber World*, 1954, **129** [4], 489–491).—An instrument is described for assessing the low-temp. characteristics of elastomers. Test specimens can be subjected to a gradual loading, and variation of compression during test and determination of recovery characteristics are possible. The instrument comprises a compression cell into which a 0·7-in. × 0·5-in. specimen is loaded; a compression is applied by means of a gear and screw and the specimen thickness at any time is measured with a crank and counter assembly. The force exerted on the pellet is measured by a strain gauge. On testing, the pellet is subjected to a number of full compression - 40 per cent. compression cycles at different platen apertures. Graphs of results indicate the effect of varying the conditioning and test temp. on a number of natural and synthetic rubber stocks. J. L. PROSSER

1157. Methods of test for chemical stoneware. British Standard Institution. (B.S. 784:1953, 19 pp.).—This first revision of the specification published in 1938 describes the various methods that have been found to give useful indications of the properties of chemical stoneware. The test methods include: (*i*) resistance to compression—a testing machine capable of exerting an axial load of 50 tons on cylindrical test pieces is used; (*ii*) transverse strength—by direct loading test apparatus; (*iii*) resistance to abrasion—by loss in weight of test sample at hourly intervals using silicon carbide as abrasive; (*iv*) specific gravity; (*v*) apparent specific gravity, apparent porosity and water absorption; (*vi*) thermal expansion—by use of a horizontal test piece to 3½ in. long heated in furnace at rate of 3° C per min., and a dial gauge registering the linear expansion relative to that of fused silica; (*vii*) resistance to thermal disruption—a new test based on lowering of transverse strength of stoneware after subjection to thermal shock; the specimens are heated in a furnace of specified temp. for 1 hr. and then quenched by immersion in water; (*viii*) acid-soluble iron; and (*ix*) resistance to acid—loss in weight of the ground sample (BS 18 to 25 mesh) after repeated treatment with H_2SO_4 - NHO_3 mixture. D. JENKINS

Optical

1158. Automatic measurement of light absorption and fluorescence on paper chromatograms. J. A. Brown and M. M. Marsh (*Anal. Chem.*, 1953, **25** [12], 1865–1869).—An automatic device for scanning paper-strip chromatograms is described. Use of

interference filters in the assembly permits the measurement of fluorescence on paper strips, and the reproducibility of results is better than that attained with other scanning devices. Sensitivity is greater on plotting per cent. transmittance rather than on plotting absorbancy. G. P. Cook

1159. **Quantitative spectrochemical analysis with the aid of a slitless spectrograph.** A. Cornu (*Cahiers de Phys.*, 1953, No. 44, 74–80).—A device for replacing the slit in a spectrograph consists of a highly polished cylindrical stainles steel rod (7 mm in diameter) arranged vertically in place of the slit. A source of light illuminating the rod at an acute angle to the collimator axis (usually $\simeq 60°$) produces a fine rectilinear image on the axis and a sharply defined spectrum, both the alignment and the definition being practically independent of the position of the source. The width of the reflected image has been deduced geometrically and the results have been checked experimentally.

Phys. Abstr.

1160. **A linear optical-density potentiometer for use in photo-electric spectrophotometry.** C. Morton (*J. Pharm. Pharmacol.*, 1954, **6** [2], 148–152).—A description is given of a linear optical-density potentiometer interchangeable with the logarithmic density potentiometer in the Unicam SP 500 and Beckman DU photo-electric spectrophotometers. The potentiometer covers the density range 0·0 to 2·0 and each equal sub-division of the scale represents a density increment of 0·001. The density is read directly on a suitable scale.

F. R. Mumford

1161. **Comparison of colorimetric results from a normal-diffuse spectrophotometer with those from a 45-degree-normal colorimeter for semiglossy specimens.** W. E. Knowles Middleton (*J. Opt. Soc. Amer.*, 1953, **43** [12], 1141–1143).—Consideration of the chromaticities and luminance factors of 97 paints (B.S. 318 C) shows that the Hardy-General Electric recording spectrophotometer frequently gives results in discord with ordinary experience when the samples are glossy or semi-glossy. G. Skirrow

1162. **Infra-red identification of materials in the fractional milligram range by means of a beam condensing system and pressed potassium bromide pellets containing the sample.** D. H. Anderson and and N. B. Woodall (*Anal. Chem.*, 1953, **25** [12], 1906–1909).—A method for the i.r. examination of samples weighing < 1 mg is described. A pair of silver chloride lenses is arranged to form a beam condensing system, having a cross sectional area < 4 sq. mm, provision being made to place the unit quickly and precisely in the sample beam. Simple micro-dies are used to form the KBr pellets, which weigh 5 mg and may contain as little as 10 μg of the test compound. G. P. Cook

1163. **New projector with many uses.** R. Dupuis (*Mikrochim. Acta*, 1953, [4], 421–425).—A projector embodying a prism is described. It can be used for transmitted, polarised, parallel, or convergent light. It is light, sturdy and easy to manipulate, and can be used as a micro-projector or as a film-strip projector. A. J. Mee

Thermal

1164. **Apparatus for measuring the quantity of heat contained in a flowing medium.** A. E. Lansner (Brit. Pat. 701,832–4, 17.10.51. Denmark 18.10.50).—The device for measuring the amount of condensate, which provides an index for the required heat measurement, comprises a scoop wheel which is retained in the filling position (by forces asymmetrical to the rotation) until the wt. of condensate flowing into the scoop is sufficient to overcome these forces and move the wheel momentarily another step to the next filling position, while discharging the amount of condensate measured. The cooling medium is a substance which, during the operation of the measuring apparatus, is, as far as possible, partly in a solid and partly in a liquid state, *e.g.*, $CaCl_2.6H_2O$, m.p. 29° C. The device for metering the condensate, located inside the closed system, is connected by a magnetic coupling to a recording device placed outside the system. J. M. Jacobs

1165. **A constant-temperature heater.** S. H. Tucker (*J. Chem. Educ.*, 1953, **30** [12], 634).—A device is described for maintaining reactions at a constant high temp. The apparatus (illustrated) consists of a tube containing the reaction mixture, surrounded by another tube containing vapour of a suitable organic solvent. A list of readily available heating-bath liquids is given whose b.p. range from 130° to 316° C. D. Bailey

1166. **Melting-point determination by the Rast capillary method, modified for the Kofler micro-heating stage.** O. Rizzolli (*Mikrochim. Acta*, 1953, [4], 401–409).—To obtain accurate results for m.p. with the Kofler micro-heating stage it is necessary to take into account that fact that the m.p. varies with the rate of heating. This can be done by extrapolating to infinitely small heating rates. To get continuous curves as simply as possible it is best to work in the region where the curve is linear, *i.e.*, at a heating rate of $< 0·3°$ per min. A. J. Mee

1167. **Distillation and determination of boiling points on a micro-scale.** W. Bodenheimer (*Nature*, 1954, **173**, 124–125).—An apparatus (with diagram) is described for a quick and accurate determination of boiling points with 1 to 3 mg of material. The apparatus can be used for fractional distillation of mixtures on a micro-scale, and the amount of distillate can be estimated. I. Jones

Electrical

1168. **A holder for platinum cathodes.** R. S. Young (*Lab. Practice*, 1954, **3** [2], 71).—The use of higher softening point plastics is preferred to wood in the construction of cathode holders. Injury to the cathode or its deposit is minimised and the bright plastic surface enables any metal from a spongy or non-adherent deposit to be readily detected. D. R. Glasson

1169. **Mercury-pool polarography. Ion-amalgam reductions occuring at surface of a large stationary mercury pool.** C. A. Streuli and W. D. Cooke (*Anal. Chem.*, 1953, **25** [11], 1691–1696).—A study of the characteristics of the mercury-pool electrode, its value as an analytical tool, and a comparison between it and the dropping-mercury electrode, is given. The polarographic waves obtained have distinct current max., whose heights are linear with respect to concn. of reducible ion; the half-wave potentials are independent of concn. For the electrode used, i_d values are between 25 and 40 times as great as for the dropping-mercury electrode,

peaks being detected at concn. of 5×10^{-7} *M*. Half-wave potentials agree, to within 0·05 V, with the $E\frac{1}{2}$ values obtained by the dropping-mercury electrode. Reproducibility of the current peaks for the same soln. is about 4 per cent. Special reference is made to the polarography of Pb^{II}, Cd^{II}, Cu^{II}, Zn^{II}, B^{III} and Tl^{I}. Other advantages of the electrode are its high hydrogen overvoltage and its wide area variation. Diffusion currents are predictable within 5 per cent. by the equations applicable to oscillographic polarography.

G. P. COOK

1170. **An improved method for routine electrolytic polishing of micro-specimens.** R. L. Hancher (*Metallurgia*, 1954, **49**, 47–51).—An almost foolproof method for routine electrolytic polishing of micro-specimens, to overcome the effects of variables, is described, and is based on voltage - resistance curves. Its functioning depends on the curve having a max. resistance corresponding to a certain voltage. The max. is found by means of a balancer circuit to indicate variation of resistance with voltage and facilitate adjustments. C. H. COWPER-COLES

1171. **Recording a.c. polarograph.** G. Buchanan and R. L. Werner (*Aust. J. Chem.*, 1953, **6** [4], 439–442).—A recording a.c. polarograph is described which can be assembled from readily available components and has advantages over the corresponding d.c. instrument in the detection of metal ions in soln. L. VALENTINE

1172. **A general purpose polarograph.** J. H. Glover and V. O. Yates (*Instrum. Pract.*, 1953, **7** [12], 963–968; Abstr. from *Bull. Brit. Sci. Instrum. Res. Ass.*, 1953, **8**, 354).—The manual polarograph described includes compensating circuits which facilitate interpretation of the current - voltage curves obtained. R. B. CLARKE

1173. **A polarographic electrolysis vessel.** A. D. Etienne (*J. Ass. Off. Agric. Chem.*, 1953, **36** [3], 1027–1029).—By using the vessel described and illustrated the complete analysis can be conducted in an atmosphere free from oxygen. The entire electrode assembly can be clamped in position with the bath and cell assembly as the only moving parts.

A. A. ELDRIDGE

1174. **Conductimetric analysis at radio-frequency: submerged-choke method.** G. G. Blake (*Analyst*, 1954, **79**, 108–109).—The rectified radio-frequency method previously described (*Brit. Abstr. C*, 1950, 303; 1951, 355) has been further developed, the conductimetric tube with external electrodes into which the sample had to be drawn being replaced by a new type of conductimetric tube that is inserted into the liquid. The system comprises a 1175 kc.p. s. oscillator controlled by a condenser, a rectifier, a radio-frequency choke and a micro-ammeter or galvanometer. Before immersion, the choke inside the conductimetric tube is connected in series with the oscillator and rectifier and the micro-ammeter is set to show no deflection. When the tube is submerged in the soln. the deflection is proportional to the conductivity of the soln. Titration graphs compare well with those plotted by either rectified radio-frequency or a.c. methods; the method is also applicable to the continuous determination and control of soln. concn.

A. O. JONES

1175. **A direct-reading inexpensive conductivity bridge.** G. W. Thiessen and J. Wertz (*Chemist Analyst*, 1953, **42** [4], 91–92).—An inexpensive direct-reading bridge suitable for rapid conductivity measurements is described. The circuit details are shown schematically and include a "standard" resistance consisting of individual resistances connected in parallel and each provided with an on - off switch so that the conductance of the standard can be varied as required. This bridge (sensitivity, 10^{-5} mhos) provides a means of obtaining approx. conductivity results rapidly and directly. The distributed capacity in the resistors can be overcome by the use of double-pole switches.

D. BAILEY

ERRATUM.—April (1954) issue, abstract 788, line 3.
For Frohmann *read* Frohman.

Translations

The following papers of interest to analytical chemists have been translated into English.

CONSULTANTS BUREAU

Copies of these papers can be obtained from Consultants Bureau, 152, West 42nd Street, New York 18, N.Y., U.S.A. Each translation costs **$7.50** and orders should state title, author(s) and English page number. The English page number is given in parentheses after the Russian page number.

These translations can also be seen in the library of the Chemical Society, Burlington House, London, W.1.

Colloid J., U.S.S.R.—

Determination of the molecular weight of casein by the light-scattering method—N. F. Dyachenko and I. N. Vlodavets, **1952**, [5], **338** (**367**).

J. Anal. Chem., U.S.S.R.—

The application of mathematical statistics in analytical chemistry—N. P. Komar, **1952**, **7** [6], 325 (361).

The ultramicro method of chemical analysis. Part I—I. P. Alimarin and M. N. Petrikova, **1952**, **7** [6], **341** (**377**).

Spectrophotometry in chemical analysis—S. I. Sinyakova and N. P. Ivanov, **1952**, **7** [6], **349** (385).

Quantitative X-ray spectrographic analysis. Part I—E. E. Vainshtein, I. D. Shevaleevsky and M. M. Kakhana, **1952**, **7** [6], **363** (**403**).

Application of the ion-exchange chromatographic method in the analysis of copper iron ceramic alloys and bronzes—D. I. Ryabchikov and V. E. Bukhtiarov, **1952**, **7** [6], **377** (**417**).

J. Appl. Chem., U.S.S.R.—

The adsorption method of separating caffeine—N. A. Izmailov, Yu. V. Shostenko and B. D. Bezugli, **1952**, [5], **543** (**611**).

Apparatus for thermal analysis—A. A. Kalandiya, **1952**, [5], **562** (**637**); *Brit. Abstr. C.*, **1953**, **233**.

DEPARTMENT OF SCIENTIFIC AND INDUSTRIAL RESEARCH

The following translations can be obtained from D.S.I.R. Technical Information and Documents Unit, Cunard Building, 15, Regent Street, London, S.W.1, at the prices shown.

Compt. Rend. Acad. Sci., U.S.S.R.—

Quick methods of microchemical analysis. A simultaneous determination of carbon, hydrogen and silicon—V. A. Klimova, M. O. Korshun and E. G. Beresnitskaya, **1952**, **84** [6], 1175. Price **32s.**

Hyg. & Sanit., U.S.S.R.—

The rapid determination of small amounts of dimethylaniline in air—N. I. Formicheva and P. A. Melnikova, **1952**, [5], 49. Price **24s.**

SCIENCE MUSEUM LIBRARY

Photo-copies of the following translation can be obtained from the Science Museum Library, South Kensington, London, S.W.7.

Compt. Rend. Acad Sci., U.S.S.R.—

A new method of simultaneous micro-determination of fluorine, hydrogen and carbon in organic compounds—N. E. Ghel'man and M. O. Korshun, **1953**, **89** [4], 685.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångstrom unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
atomic	at.
boiling-point	b.p.
British thermal unit	B.Th.U.
calculated	(calc.)
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
critical	crit.
crystalline, crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c.p.s.
decompos -ing, -ition	(decomp.)
density	ρ
density, relative	d or wt. per ml
derivative	deriv.
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
electron-volt	eV
equivalent	equiv.
experiment, -al	expt.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
horse-power	h.p.
hour	hr.
hydrogen ion concentration	$[H^{\cdot}]$
hydrogen ion exponent	pH
inch	in.
indefinite	indef.
infra-red	i.r.
insoluble	insol.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim -um, -a	max.
melting-point	m.p.
microgram	μg
micro-litre	μl
micron	μ
milliampere	mA
milligram	mg
millilitre	ml
millimetre	mm
millivolt	mV
minimum	min.
minute (time)	min.
molar (concentration)	M
molecul -e, -ar	mol.
normal (concentration)	N
number	no.
observed	(obs.)
organic	org.
ounce	oz.
part	pt.
patent	pat.
parts per million	p.p.m.
per cent. wt. in wt.	per cent. w/w
per cent. wt. in vol.	per cent. w/v
per cent. vol. in vol.	per cent. v/v
potential difference	p.d.
pound	lb
precipitate	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	$n\lambda^t$
relative humidity	R.H.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]\lambda^t$
square centimetre	sq. cm
standard temperature and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition the following symbols are used—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

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ANALYTICAL ABSTRACTS

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